REPORT ON AN INVESTIGATION OF PROBLEMS IN THE MEASUREMENT OF THIN EVAPORATED METAL FILMS ON MYLAR

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SPACE-GENERAL CORPORATION
El Monte, California

Report No. SGC 220S-3
Contract NAS 1-1732

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June 1964

For

NASA/LANGLEY RESEARCH CENTER

SPACE GENERAL TIRE GENERAL

REPORT ON AN INVESTIGATION

OF PROBLEMS IN THE MEASUREMENT

OF THIN EVAPORATED METAL FILMS ON MYLAR

Report No. SGC 220S-3

Prepared Under

Inflatable Micrometeoroid Paraglider Program

Contract NAS 1-1732

For

NASA/LANGLEY RESEARCH CENTER

Prepared by:

Earl G. Blackwell

E. G. Blackwell
Principal Investigator

Approved by:

James D. Campbell

J. D. Campbell
Project Engineer

SPACE-GENERAL CORPORATION

9200 East Flair Drive
El Monte, California

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1.0 INTRODUCTION

1.1 BACKGROUND INFORMATION

Under a recent contract from NASA, Space-General Corporation, El Monte, California, fitted a pneumatically extended paraglider with capacitance type micrometeoroid detectors covering the wing membranes.

These detectors, which were developed by NASA, Langley Research Center, are constructed of a multi-layer assembly of vacuum metalized polyester film; the alternate layers of metal and mylar forming the capacitive elements. The sensors are constructed so as to have three layers of .00025" metalized mylar (1/4 mil) comprising the outer surface of the detectors and two layers of .0005" metalized mylar below. The bottom layer of mylar is metalized on both sides; thus, three capacitors are formed.

In operation the capacitor complex is charged to about 40 volts each and upon a meteoric hit the capacitor or capacitors penetrated will momentarily discharge indicating a hit. The depth of penetration is, roughly, a function of particle energy and is indicated by the number of capacitors penetrated. The degree of discharge is a rough measure of particle size.

1.1.1 The optimum thickness of the metal film has been estimated at about one-tenth the dielectric thickness. As sensor performance may depend on the thickness of the evaporated metal film, the thickness becomes a control parameter in the construction of micrometeoroid detectors of the capacitance type.

1.1.2 The thickness range encountered in metal films on this type of sensor may be considered to range from 25 millionths to 50 millionths of an inch. This would correspond to the optimum metal depositions on 1/4 mil mylar and 1/2 mil mylar, respectively.

1.1.3 This particular range of thickness creates a difficulty in presently available techniques for conveniently monitoring the thickness of these metallic films, because of the following general reasons:

a. Optical transmission ceases around 5 micro inches.

b. Direct overall measurement by mechanical, electronic and optical micrometers is encumbered by relatively gross inhomogeneities in the mylar substrate thickness.

c. Interferometry is unsuitable as the base material isn't sufficiently flat to give a satisfactory fringe pattern.

Previously these film determinations were made by direct measurement of photomicrographs of microtome cross sections enlarged about two thousand (2000) times.

1.2 PROGRAM OBJECTIVES AND APPROACH

1.2.1 It was the purpose of this program to investigate the application of commercially available non-destructive thickness indicators and feasible laboratory techniques to the problems of measurement of these thin vacuum deposited metallic films for in-process deposition control and/or non-destructive inspection prior to assembly.

1.2.2 The proposed approach consisted of the following areas of investigation:

a. Initial measurement of thickness by microscope micrometer of microtome cross sections of several specimens taken from different vendor's samples of vacuum metalized mylar.

b. Attempt to calibrate and/or correlate these data with measurements made with:

(1) Beta Ray backscatter instruments
(2) R.F. Eddy-Current instruments
(3) Any other applicable instruments available

c. Obtain chemical gravimetric analysis on specimen taken from vendor's samples and correlate this surface density information with other data.

d. Make laboratory potentiometric resistive measurements of long thin specimen samples to determine effective sheet resistivity.

e. Correlate the above data in a presentation to show problems of data scatter, deviations from theoretical extensions of bulk wrought characteristics.

f. Make recommendations for process control in:

(1) Vacuum deposition processes

(2) Inspection techniques for sensor materials

(3) Most applicable type of commercial instruments

(4) Significant problem areas of these approaches

1.2.3 As information regarding vendor deposition processes on their supplied samples such as, rate, source temperature, substrate temperature, and vacuum, are not available, it will be necessary to generalize in certain areas of density-thickness correlations, and thickness-resistivity correlations to the extent that they are assumed to be typical evaporated metallic films and their trends should be reflected in similar metallic films which have been vacuum deposited.

2.0 CONCLUSIONS AND RECOMMENDATIONS

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The conclusions from the data accumulated in this investigation are based on relatively few samples of limited distribution; however, the data from this work does clearly indicate many of the problem areas in the measurement of thin evaporated metal films on mylar.

From the plots of thickness vs. sheet resistivity and thickness vs. mass/unit area, a predictable correlation is shown with the copper and gold samples. It is also seen that these functions are considerably displaced from wrought characteristics. The aluminum showed a great deal of scatter from any predictable relationship.

Information regarding the manufacturers process control parameters were purposely ignored in order to make an unbiased study of thickness measurements of these metallic films, independent of process.

Individual thickness determinations measured directly under the microscope are subject to considerable scatter from the average thickness as they are confined to a single point. Therefore, several cross sections should be averaged if direct linear thicknesses are made. The indirect thickness determinations showed less scatter, as their measurements involved a finite area, hence more nearly a typical average.

It is indicated from this study that non-destructive instruments whose measurement principle is based on resistivity or comparative mass per unit area may be used for thickness indications of copper or gold evaporated films on mylar provided these instruments are calibrated by samples of known thickness and identical deposition processes.

As no definite correlation of thickness to resistivity or apparent area density was found with aluminum, it is recommended that thickness determinations be made by direct methods, such as the measurement of microtome cross sections with an image splitting eyepiece.

Author

Existence of systematic errors due to deformation of the metallic film by the microtome blade is neither confirmed nor refuted by this study.

Areas needing future study indicated by this program are:

(1) Correlation of mass/unit area and sheet resistivity vs. thickness to dependence on deposition process variables.

(2) Studies paralleling this one, but with more samples having a wider parameter distribution, especially with thicknesses below one micron.

3.0 INVESTIGATION TECHNIQUE AND APPROACH

3.1 SAMPLE TAKING PLAN

3.1.1 A definite order was established, where applicable, for the sample removal from various vendors materials, in order to get an even distribution of characteristics across the width of a given sample roll.

The aluminum and gold samples were of ample size for the full scale samples to be removed; the copper samples, however, which arrived later in the program, were of various sizes and shapes, requiring a slightly modified sample taking plan.

The diagrams for these sample removal plans are shown in figures 1, 2 and 3.

The purpose for the different shaped samples, and the code letter identification is shown below:

3.1.2

Sample Types	Size	Use
U	7" x 6"	Eddy current, Beta Ray backscatter and reserve sectioning
V	36" x 1/4"	Resistivity
W	36" x 3/4"	Resistivity
Z	1/2" x 1/2"	Microtome Sectioning
S	3" x 3"	Square, for Gravimetric Analysis

Material Identifications

A - Aluminum - Vapor Deposited

G - Gold, 24K, Vapor Deposited

C - Copper, Vapor Deposited

Roll Identifications

1. Hastings & Co. Roll #6647 (15345) M2S, nominal 1/2 mil mylar, al thick .025 mil (not used)

2. Hastings & Co. Roll #6647 (15793) MlS, nominal 1/2 mil mylar, al thick .025 mil
3. Hastings & Co. Roll #13307 (17676) MlS, nominal 1 mil mylar, al
4. Hastings & Co. Roll #13307 (17674) MlS, nominal 1 mil mylar, al
5. Hastings & Co. Roll #13307 (17675) MlS, nominal 1 mil mylar, al
6. Hastings & Co. Roll #(unknown) MlS, nominal 1 mil mylar, gold, (ident. "A")
7. Hastings & Co. Roll #(unknown) MlS, nominal, 1 mil mylar, gold, (ident. "B")
8. Hastings & Co. Roll #(unknown) MlS, nominal, 1 mil mylar, gold, (ident. "C")
9. American Lamotite .35 mil al foil laminated to .25 mil mylar (not used)
10. G. T. Schjeldahl Company - Roll number (unknown) 2 mil mylar, copper
11. G. T. Schjeldahl Company - Roll number (unknown) 1 mil mylar, copper
12. G. T. Schjeldahl Company - sample 2 mil mylar, copper

3.2 MICROTOME TECHNIQUE

3.2.1 During a recent micrometeoroid sensor fabrication program, microtome cross sections of the vacuum metalized mylar films were used for metal film thickness determinations of the basic materials prior to assembly, and after assembly to view the composite laminations of an assembled multilayer sensor. This work was done by an outside laboratory possessing a microtome machine.

3.2.2 In the current program, arrangements were made with a vendor to loan Space-General Corporation a microtome of the freezing type. Generally the performance of this machine was quite satisfactory and proved to be an easy way of obtaining the required cross sections.

3.2.3 In operation a small sample about 1/8" by 1/2", of the metalized film to be cross sectioned is frozen on edge in a distilled water matrix onto a small brass disc. This disc is then mounted in a chuck type assembly which drives the water encapsulated sample passed a heavy rigid knife in a reciprocating motion. The specimen is automatically advanced each cycle to a preset increment, making slices about 4 to 8 microns thick. The thin slice is then "captured" as it rests on the blade by bringing a microscope slide in contact with the ice fragment. Upon contact with the room temperature glass slide the ice melts adhering to the slide - a microscope examination will confirm whether a satisfactory sample has been obtained.

3.2.4 A satisfactory sample is considered as one of smooth continuous filament with little or no spiral.

3.2.5 The slicing technique developed consisted of rotating the brass sample holder until the length of the specimen is almost perpendicular to the edge of the microtome blade. Rough cutting is then done to remove superfluous ice until the full length of the sample is exposed. The sample and holder is rotated to place the length of the sample almost parallel to the microtome. This cutting angle reduces the curl to almost zero, allowing the sample to lie flat on the glass slide, facilitating an edge-on view of the metal coating. A photograph showing curling when the specimen is perpendicular to the knife is included.

3.2.6 It is also important to position the sample with respect to the knife so that the metalized side is first to encounter the blade; otherwise, if the metal film is at the rear surface of the mylar, the blade will strip off the thin metal film. It was noted that the grain of the roll should be parallel to the direction of slicing for "clean" cuts.

3.2.7 After a satisfactory specimen had been obtained, it is then allowed to dry and permanentized by placing a small drop of Canadian Balsam on the center of the slide and lightly pressing on a cover glass.

3.2.8 At this point, the slide is placed under a camera equipped microscope and a typical cross section view is found and photographed. The primary purpose of the photograph is to provide a permanent record.

3.2.9 Linear thickness measurements were performed under the microscope using a bi-color image splitting eyepiece micrometer. In use, the image appears as black and white when the micrometer scale is set to zero. As the micrometer is turned, the single black image becomes a red and green image whose separation is proportional to the dial setting. To measure the width of an object, the micrometer dial is set to zero, then turned until the two colors are just separating. The dial reading is calibrated by reference to a stage micrometer. These readings were found to be repeatable to about 5 percent.

3.2.10 A difficulty encountered with this approach was smearing of the metallic film across the mylar by the wiping action of the microtome blade. It was noted, when clearly visible, that this effect could give as much as a 40 percent to 80 percent increase to the apparent film width. With this effect in mind, precautions were taken to find the thinnest undistorted location on the microscope specimen for the actual measurement. Six specimens were taken from each sample roll to give measurement scatter information and a good mean thickness value. Photographs of cross sections typical of each sample roll are included in this report.

3.2.11 The results of these measurements are tabulated in Table I.

3.3 SHEET RESISTIVITY MEASUREMENTS

3.3.1 The information regarding sheet resistivity was obtained by using long thin specimen and finding the total resistance; the ohms/square being calculated by this total resistance and the strip dimensions, i.e.

$$R_s = \frac{R_t\,(W)}{L} \quad \text{ohms/square}$$

where R_s is the sheet resistivity in ohms/square, R_t is the total strip resistance, W is the strip width, and L the strip length.

3.3.2 These measurements were made using a "Leeds and Northrup Resistance" bridge and electronic null indicating galvanometer. These readings were usually quite repeatable to better than one percent. A tabulation of these readings is given in Table 2.

3.4 BETA RAY BACKSCATTER MEASUREMENTS

3.4.1 An instrument of the Beta Backscatter type was borrowed from a local representative. The trade name of this particular instrument is MICRODERM.

In principal, these instruments are nuclear radiation counters, which may be fitted with a number of different radioactive sources. Several samples of known thickness are used to calibrate the instrument and establish a curve for a particular metal on a particular substrate material. In operation, the calibration samples are placed in position over a small aperture, immediately below which is a small point radioactive source of some specified Beta energy. Slightly back of the source, which radiates only forward, is a special mica windowed geiger tube, but which reads only backscattered rays. The thinner and less dense coatings usually requiring a lower particle energy for resolution of very small thickness and/or mass/unit area differences.

Next a relatively infinite thick sample of the backing material (.050" mylar) is read. The amount of accumulated reading for this backing material is then biased out; that is, set for zero reading. Full scale may have to be slightly readjusted to 100%. The instrument now reads only the differential backscatter between the metalized mylar and the mylar alone.

Several other thinner samples may then be read and a calibration curve plotted. Once the microderm has been set up, it requires only about 60 seconds to read any sample thickness of the same materials.

According to the manufacturer, this instrument will measure the thickness of any material on any other material, as long as there is a sufficient difference in atomic number. This technique of measurement, of course, responds to mass per unit area and is an indirect measurement of thickness. The curves obtained with gold are shown in Figure 10.

The aluminum samples did not give sufficient reading for a full scale setting. Even after an eight minute reading, the thickest aluminum samples (66 micro inches) would only register a 6% differential reading. This was using the lowest energy beta source, Carbon 14. According to the instruction manual, aluminum and plastic were sufficiently different in atomic number to be measurable. Perhaps there was a lower restriction on mass per unit area.

Only one copper sample was available concurrent with the microderm; therefore, no curve was made. The copper sample tried, however, did give a good 100% reading. This sample later turned out to be the thinnest of the copper samples. It is, therefore, assumed that satisfactory operation can be obtained with both gold and copper on mylar in these thickness ranges.

On the work in this report, a Carbon 14 source of 0.11 Mev Beta particles was used, which was the lowest energy source with the instrument.

In principle, when calibrating the Microderm, a reading is taken for, say one minute, of the thickest gold on mylar sample. An adjustment is then made to make the meter on the instrument read full scale or 100%.

3.5 CHEMICAL GRAVIMETRIC ANALYSIS

3.5.1 The mass per unit area of the metalized mylar samples was determined by gravimetric analysis from a local metallurgical laboratory. In these determinations, three samples 3" by 3" of each roll of material were cut at evenly spaced intervals across the roll width in order to establish a typical average sample density. The results of this work are tabulated in Table 3.

3.6 EDDY CURRENT MEASUREMENTS

3.6.1 The thickness of a coating of conducting material on a non-conductor may be ascertained by two different approaches based on the eddy current principle.

On technique uses the skin effect depth as a function of frequency, the other measures the eddy current loading through mutual coupling. No instrument using the skin effect could be located which would operate in the required thickness range.

3.6.2 The Instrument Division of the Budd Company, however, claimed to have operated their instrument (eddy current loading effect) successfully in the 100 micro-inch range previously, and agreed to load us such an instrument (Radac 210) for our measurements. It was decided that due to the lack of time available, faster results could be obtained by taking the samples back East to the plant rather than ship the instrument here to Space-General. These measurements were then made by their Applications Department.

3.6.3 This instrument's principle is, of course, reliant on sheet resistivity and would be dependent on the consistency of this parameter with the reciprocal of thickness. This instrument's relative scale readings are similar to the microderm in that they must be calibrated with samples of known thickness. These percent scale readings are shown in Figure 11.

4.0 DISCUSSION OF RESULTS AND DATA CORRELATION

4.1 GRAPHIC PLOTS AND TABULATIONS

The graphic plots of the following relationships are shown with their respective figure designations:

4.1.1 Sheet Resistivity vs. Thickness

- a. Aluminum on mylar — Figure 4
- b. Copper on mylar — Figure 5
- c. Gold on mylar — Figure 6

4.1.2 Mass per Unit Area vs. Thickness

- a. Aluminum - mylar — Figure 7
- b. Copper - mylar — Figure 8
- c. Gold - mylar — Figure 9

4.1.3 Beta Backscatter Curve

- a. Gold - mylar — Figure 10

4.1.4 Tabulations

- a. Table 1 - thickness
- b. Table 2 - sheet resistivity
- c. Table 3 - Gravimetric analysis

TABLE 1

MICROTOME CROSS SECTION MEASUREMENTS

Methods Used: Bicolor Image Splitting Micrometer at 1500 x (readings repeat at 4%)

Aluminum		Gold		Copper	
Sample	Thickness Micro inches	Sample	Thickness Micro inches	Sample	Thickness Micro inches
2AZ1	60.5	6GZ1	60.5	11CZ1	61.5
2AZ2	62.2	6GZ2	57.0	11CZ2	65.0
2AZ3	65.7	6GZ3	46.0	11CZ3	68.3
2AZ4	67.8	6GZ4	47.6	11CZ4	66.6
2AZ5	72.0	6GZ5	52.3	11CZ5	57.9
2AZ6	72.0	6GZ6	55.4	11CZ6	59.0
Mean	66.6	Mean	52.3	Mean	63.0
3AZ1	52.3	7GZ1	49.3	10CZ1	196.
3AZ2	56.3	7GZ2	54.3	10CZ2	173.
3AZ3	57.0	7GZ3	82.3	10CZ3	150.
3AZ4	50.4	7GZ4	75.4	10CZ4	108.
3AZ5	50.4	7GZ5	64.0	10CZ5	134.
3AZ6	48.5	7GZ6	85.0	10CZ6	165.
Mean	53.2	Mean	68.4	Mean	154.
4AZ1	59.0	8GZ1	79.5	12CZ1	54.7
4AZ2	48.1	8GZ2	90.2	12CZ2	55.1
4AZ3	46.0	8GZ3	95.4	12CZ3	57.9
4AZ4	49.3	8GZ4	82.1	12CZ4	46.0
4AZ5	62.2	8GZ5	88.5	12CZ5	65.7
4AZ6	49.3	8GZ6	104.5	12CZ6	50.6
Mean	52.3	Mean	89.0	Mean	53.4
5AZ1	59.0			13CZ1	92.2
5AZ2	48.1			13CZ2	88.3
5AZ3	48.1			Mean	90.2
5AZ4	49.3				
5AZ5	46.0				
5AZ6	59.0				
Mean	51.6				

TABLE 2

SHEET RESISTIVITY; THIN METALLIC FILM ON MYLAR

Measurement Method: Resistance of Long Narrow Strip

$$R_s = \frac{R_t\,(W)}{L}, \text{ where } R_s = \text{Sheet resistivity}$$

R_t = Total strip resistance

W = Strip width

L = Strip length

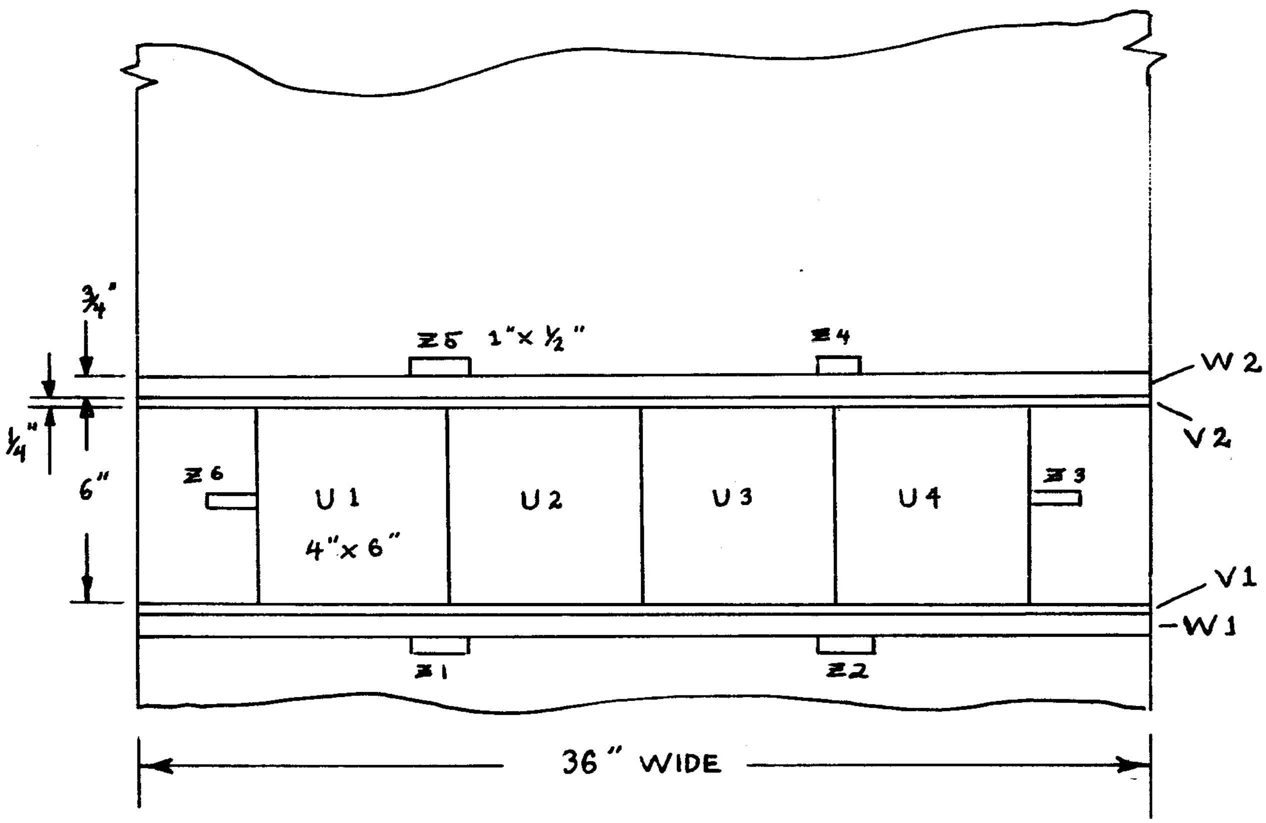
Aluminum		Gold		Copper	
Sample	Resistivity ohms/sq.	Sample	Resistivity ohms/sq.	Sample	Resistivity ohms/sq.
2AV1	.4730	6GV1	.3960	11CV1	.0552
2AV2	.4763	6GV2	.3893	11CV2	.0607
2AW1	.4855	6GW1	.3852	11CW1	.0563
2AW2	.5060	6GW2	.3865	11CW2	.0654
Mean	.4852	Mean	.3892	Mean	.0593
3AV1	1.132	7GV1	.2123	10CV1	NG
3AV2	1.078	7GV2	.2040	10CV2	.0733
3AW1	1.098	7GW1	.2125	10CW1	.0463
3AW2	1.088	7GW2	.2130	10CW2	.0139
Mean	1.099	Mean	.2102	Mean	.0334
4AV1	.5265	8GV1	.1518	12CV1	.0546
4AV2	.4920	8GV2	.1580	12CV2	.0670
4AW1	.5035	8GW1	.1596	12CW1	.0612
4AW2	.4780	8GW2	.1586	12CW2	.0682
Mean	.5000	Mean	.1570	Mean	.0628
5AV1	.5370				
5AV2	.5350				
5AW1	.5025				
5AW2	.5102				
Mean	.5212				

TABLE 3

Grams Weight/Square inch; Thin Metallic Films on Mylar

Measurement Method: Chemical Gravimetric Analysis

Aluminum		Gold		Copper	
Sample	Milligrams/in^2	Sample	Milligrams/in^2	Sample	Milligrams/in^2
2AS1	0.312	6GS1	2.19	10CS1	18.7
2AS2	0.256	6GS2	2.29	10CS2	18.6
2AS3	0.211	6GS3	2.34	10CS3	14.7
Mean	0.260	Mean	2.27	Mean	17.3
3AS1	0.067	7GS1	4.15	11CS1	4.00
3AS2	0.178	7GS2	4.22	11CS2	4.17
3AS3	0.133	7GS3	4.23	11CS3	4.68
Mean	0.126	Mean	4.20	Mean	4.28
4AS1	0.224	8GS1	6.02	12CS1	2.29
4AS2	0.312	8GS2	6.37	12CS2	2.08
4AS3	0.244	8GS3	6.54	12CS3	3.04
Mean	0.267	Mean	6.31	Mean	2.47
5AS1	0.378				
5AS2	0.356				
5AS3	0.255				
Mean	0.329				



GENERAL LOCATION OF SAMPLES

Sample Identification:

1st digit - Roll identification
2nd digit - Material identifier
3rd digit - Type of sample
4th digit - Sample no.

ALTERNATE SPECIMEN CUTTING PLAN
FOR ROLLS NARROWER THAN 36"

for dimensions see figure 1

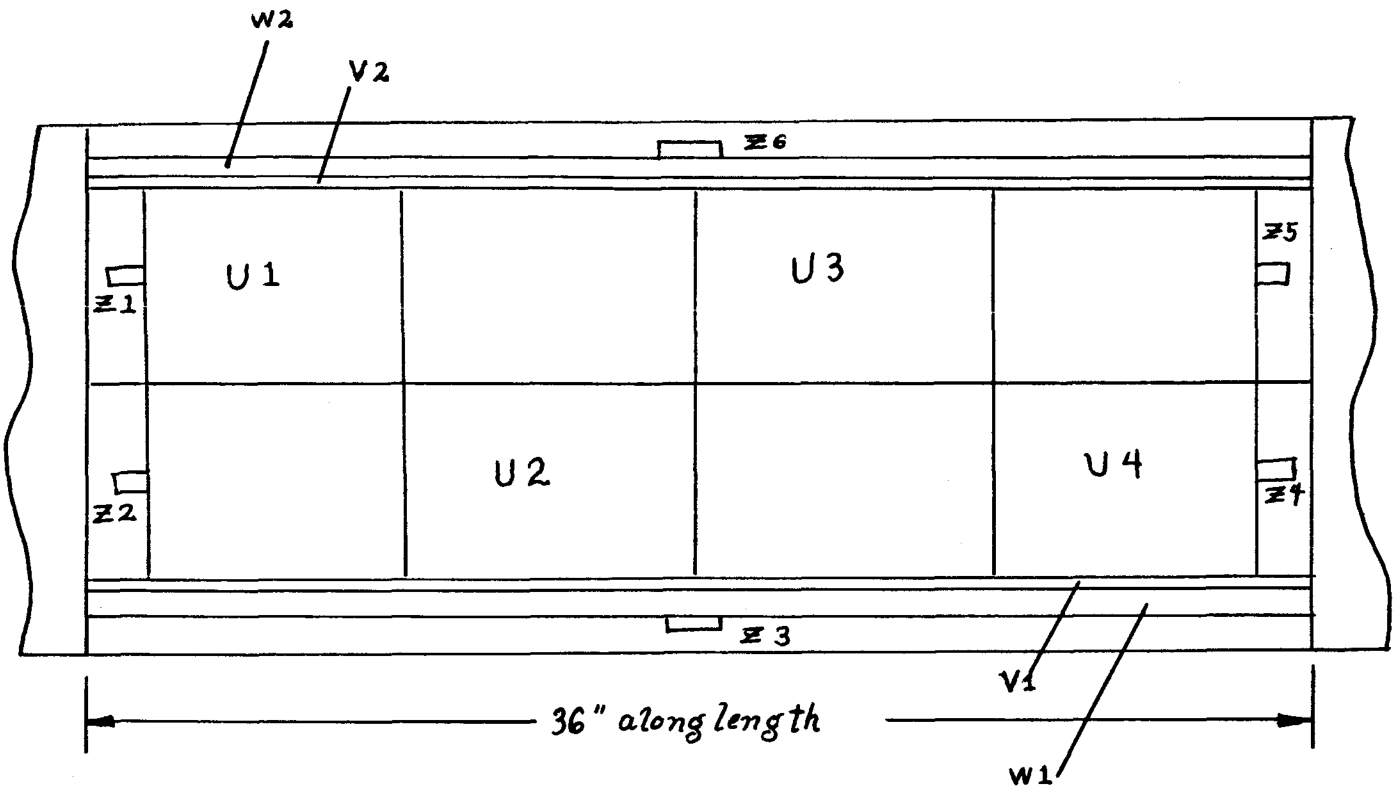
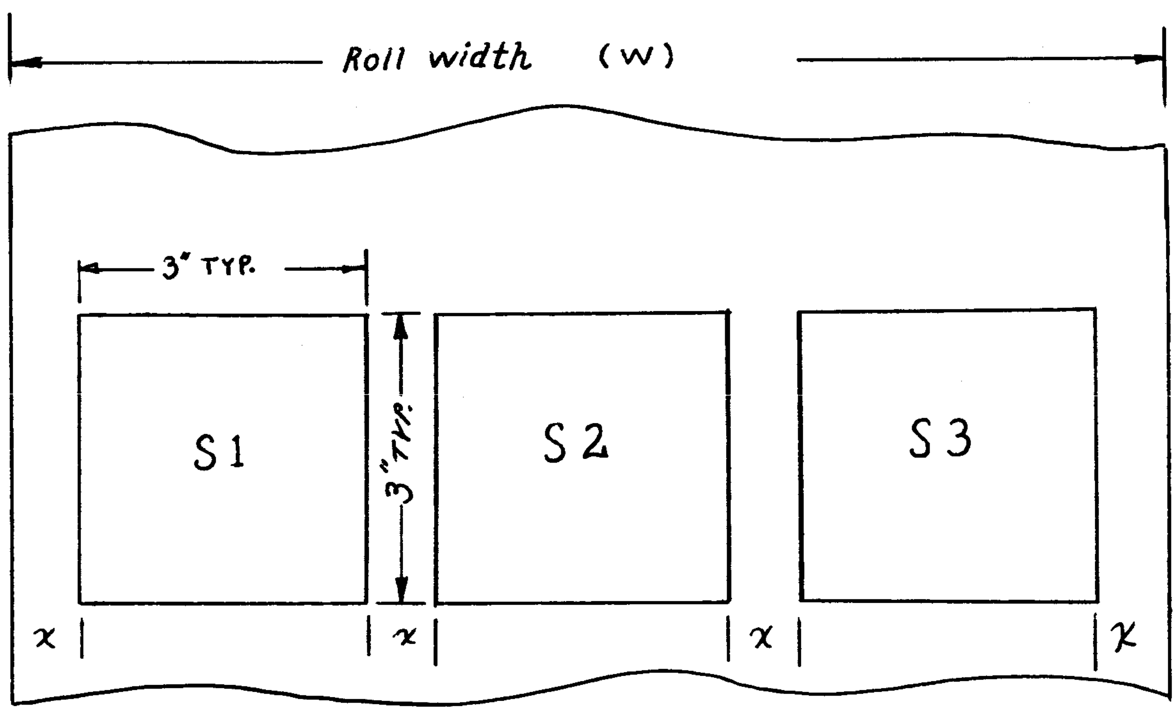


Figure 2

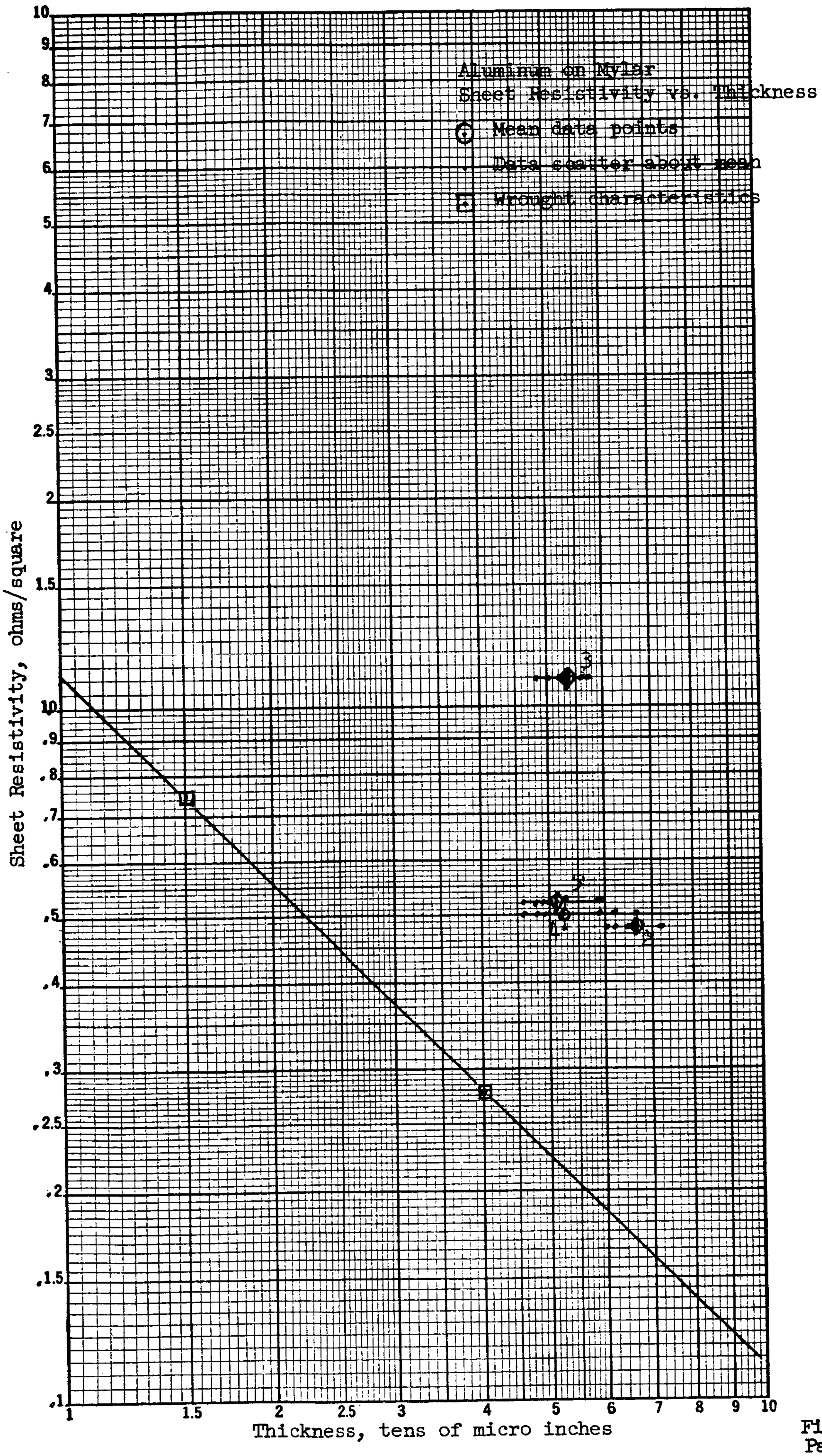
SAMPLE TAKING PLAN FOR GRAVIMETRIC ANALYSIS



Note: "x" is defined as:

$$x = \frac{W - 9}{4} \quad W = \text{Roll width}$$

Figure 3



Aluminum on Mylar
Sheet Resistivity vs. Thickness
Mean data points
Data scatter about mean
Wrought characteristics
Sheet Resistivity, ohms/square
Thickness, tens of micro inches

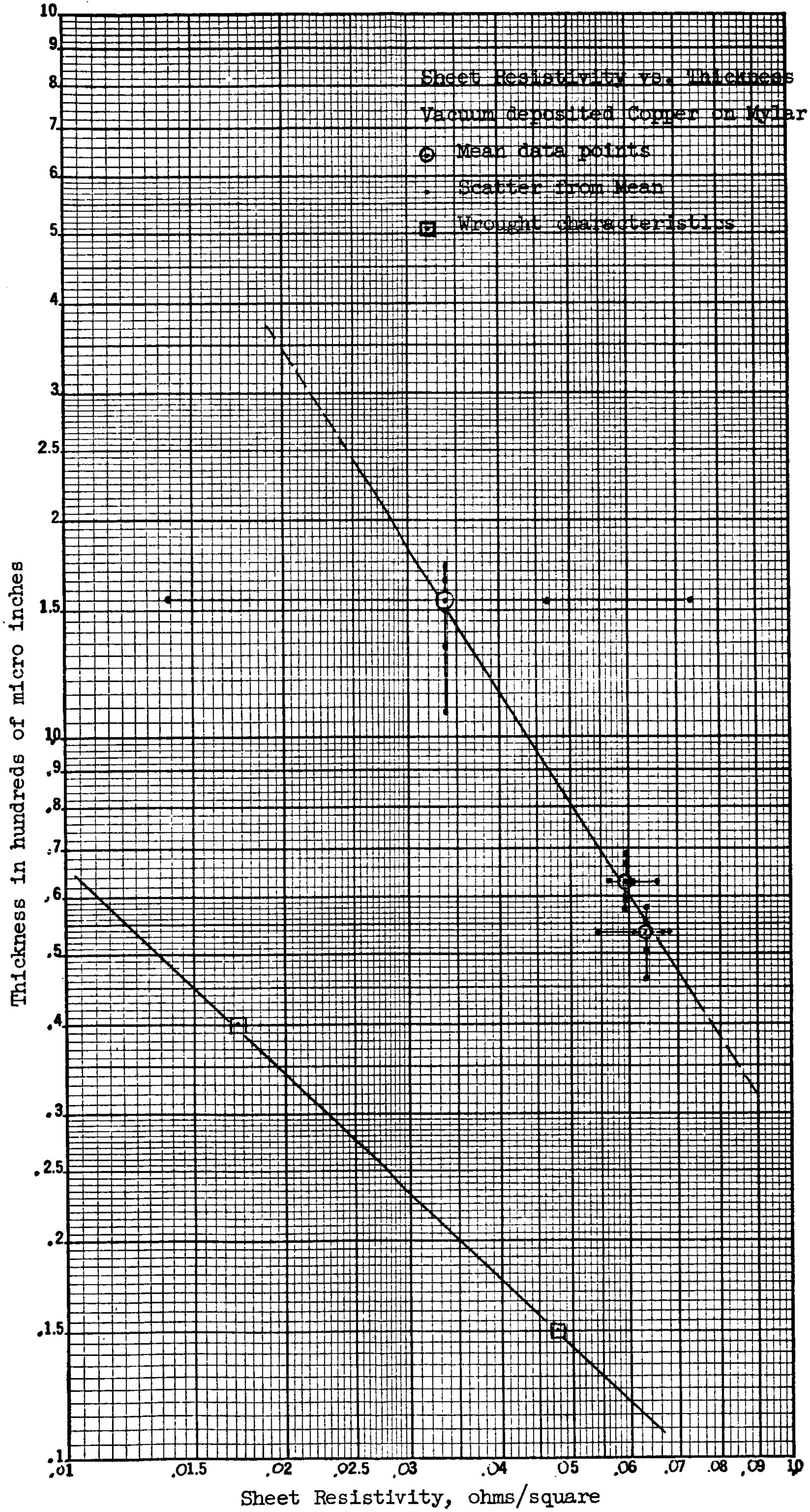

Sheet Resistivity vs. Thickness
Vacuum deposited Copper on Mylar
Mean data points
Scatter from Mean
Wrought characteristics
Thickness in hundreds of micro inches
Sheet Resistivity, ohms/square

Figure 5

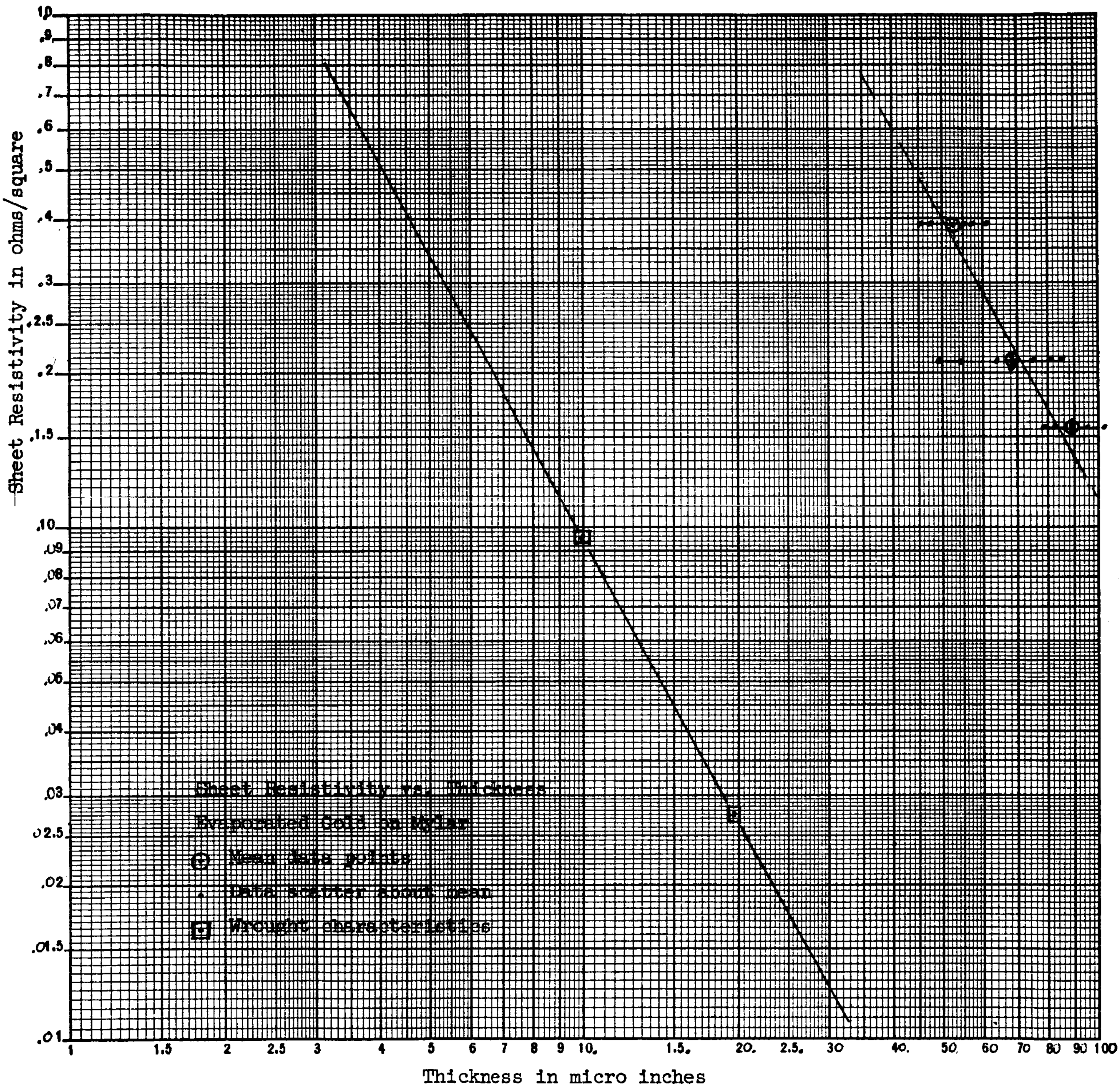


Figure 6
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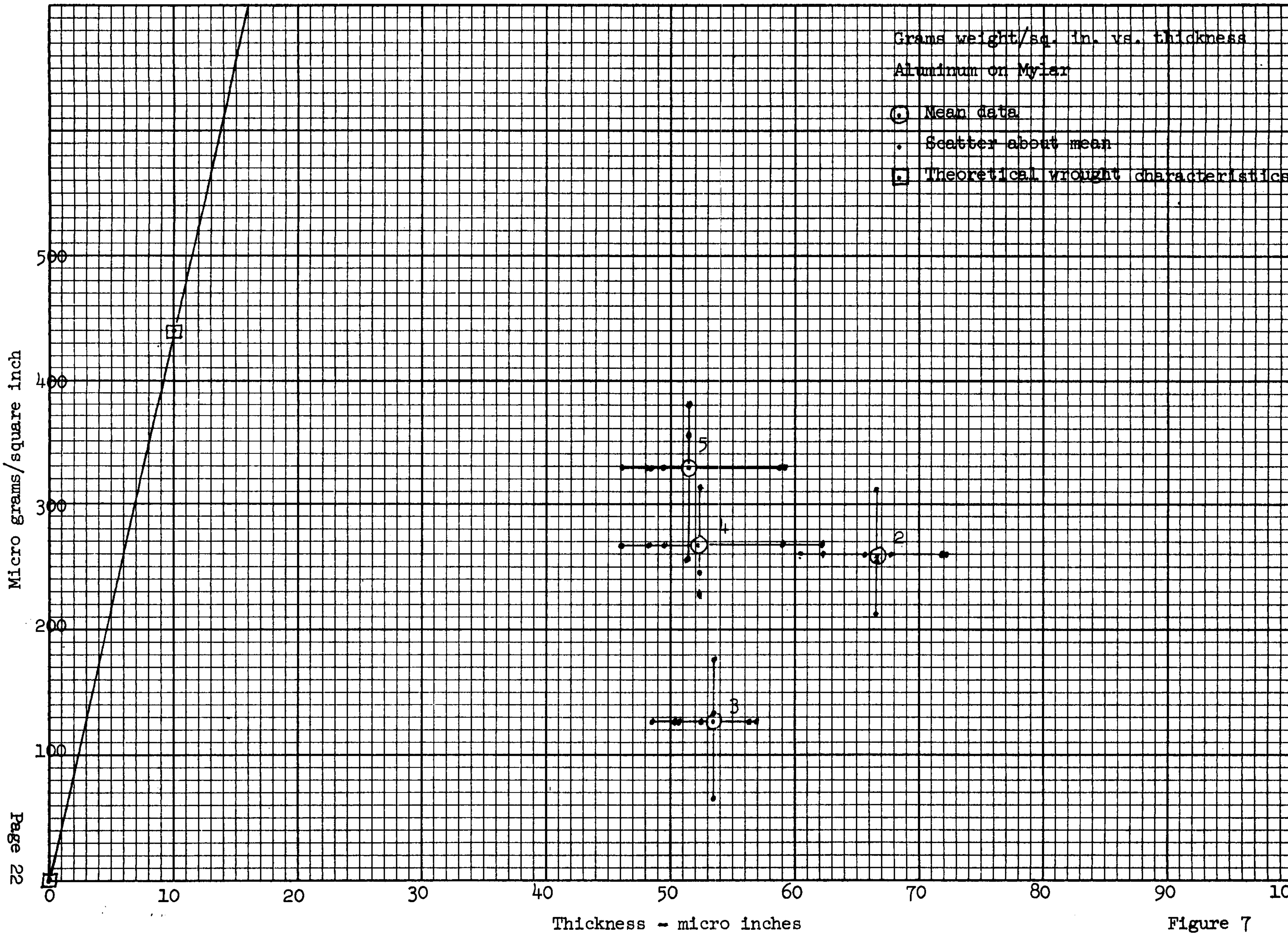

Grams weight/sq. in. vs. thickness
Aluminum on Mylar
Mean data
Scatter about mean
Theoretical wrought characteristics
Micro grams/square inch
500
400
300
200
100
0
10
20
30
40
50
60
70
80
90
100
5
4
2
3
Thickness - micro inches

Figure 7

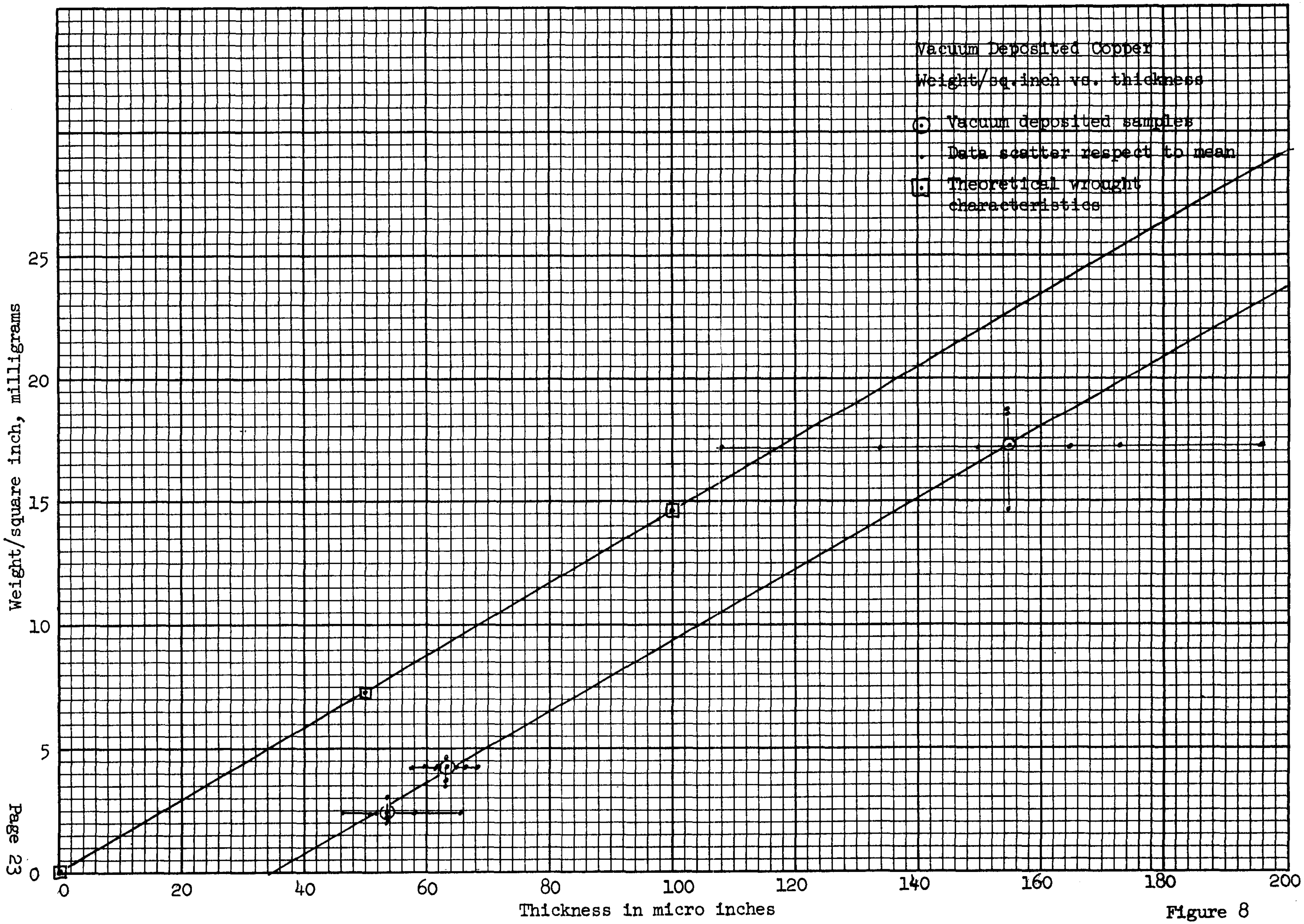

Vacuum Deposited Copper
Weight/sq.inch vs. thickness
Vacuum deposited samples
Data scatter respect to mean
Theoretical wrought characteristics
Weight/square inch, milligrams
0
5
10
15
20
25
0
20
40
60
80
100
120
140
160
180
200
Thickness in micro inches

Figure 8

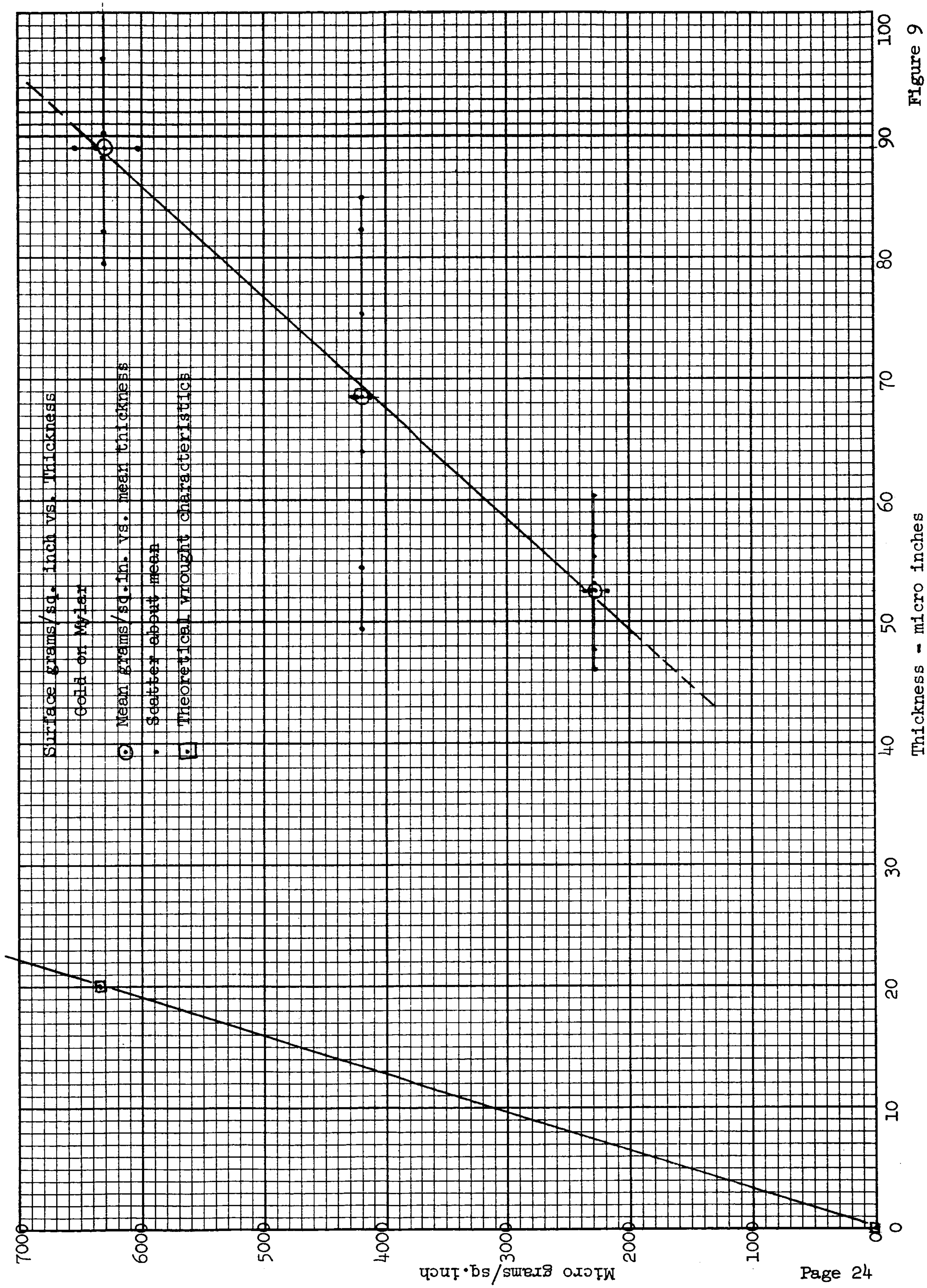

Surface grams/sq. inch vs. Thickness
Gold on Mylar
Mean grams/sq.in. vs. mean thickness
Scatter about mean
Theoretical wrought characteristics
Thickness - micro inches
Micro grams/sq.inch
0
10
20
30
40
50
60
70
80
90
100
1000
2000
3000
4000
5000
6000
7000

Figure 9

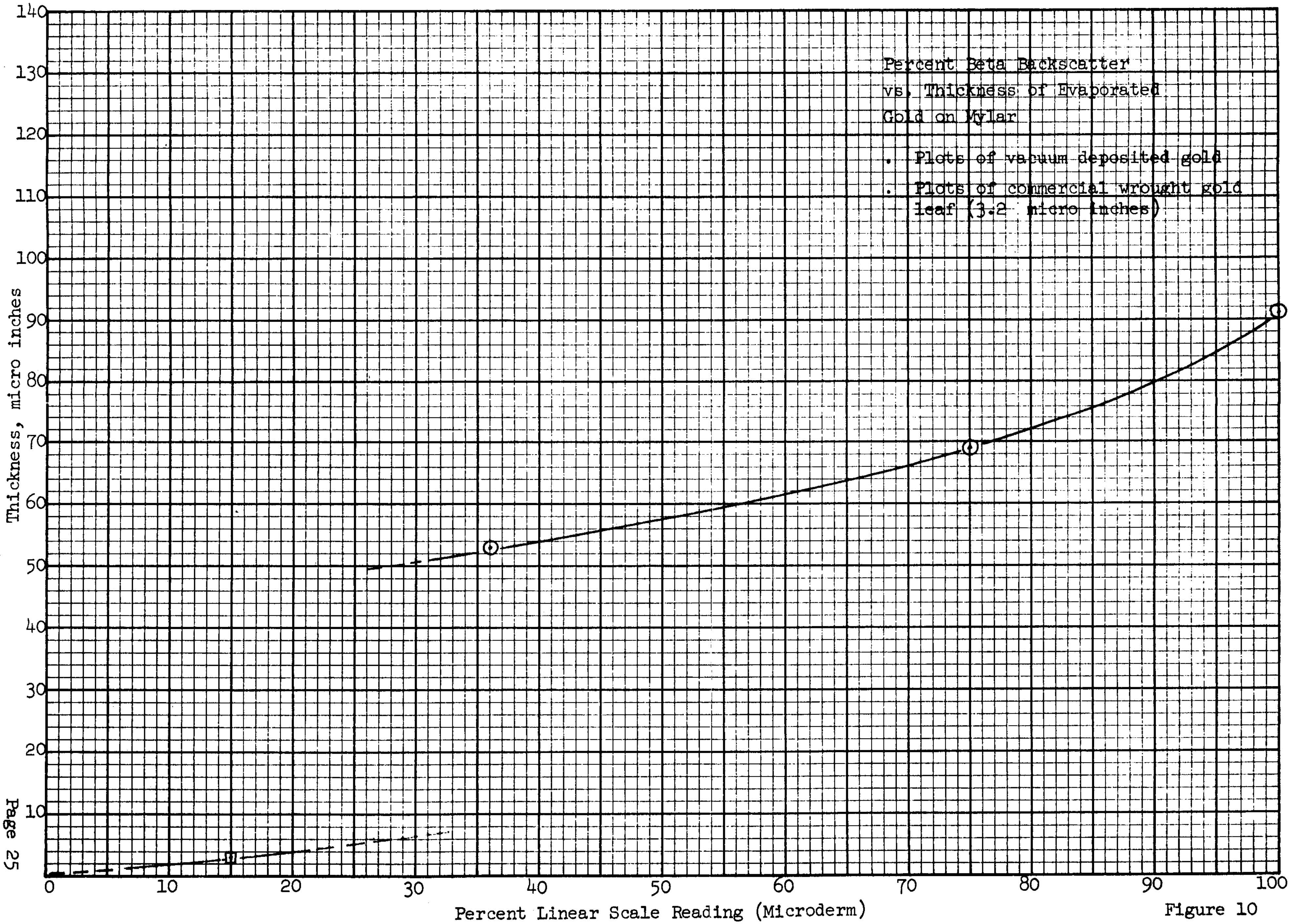

Percent Beta Backscatter
vs. Thickness of Evaporated
Gold on Mylar
• Plots of vacuum deposited gold
· Plots of commercial wrought gold leaf (3.2 micro inches)
Thickness, micro inches
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
10
20
30
40
50
60
70
80
90
100
Percent Linear Scale Reading (Microderm)

Figure 10

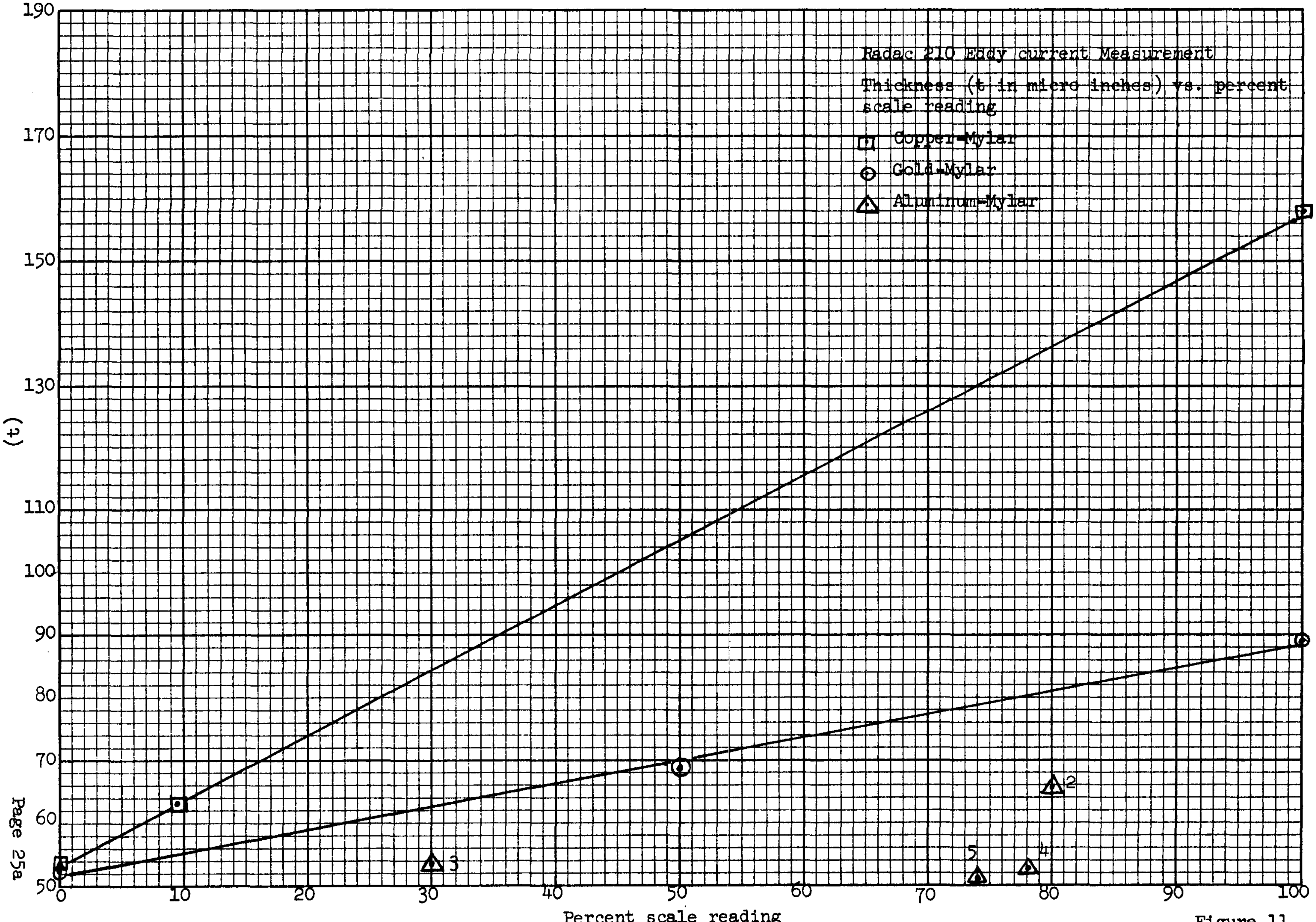

Radac 210 Eddy current Measurement
Thickness (t in micro inches) vs. percent scale reading
Copper-Mylar
Gold-Mylar
Aluminum-Mylar
(t)
190
170
150
130
110
100
90
80
70
60
50
0
10
20
30
40
50
60
70
80
90
100
2
3
4
5
Percent scale reading

Figure 11

4.2 DISCUSSION OF DATA CORRELATION

Upon examination of the aforementioned graphs, the problem of correlation of apparent area density and sheet resistivity with thickness becomes obvious. This is most severely pronounced with the aluminum samples. The copper and gold mylar plots begin to show expected trends of approximate slope but considerably displaced from wrought characteristics. Scattering of individual data points are shown in respect to the mean plots on the resistivity and mass/square inch vs. thickness graphs only. Comparatively good results were obtained with the gold and copper samples on the Radac 201, and the Beta gage Microderm. However, it was noticed that the Beta Backscatter curve, when extrapolated to zero scale reading, did not correspond to zero thickness as would be expected. The reason for this anomaly is not clearly understood. It could be either an error in the cross-section measurements or a non-linearity of thickness vs. density.

An interesting note is that a sample of commercial gold leaf (reputed to be about 3.2 micro-inches) plotted considerably displaced below the evaporated film curve.

This latter supposition is partially supported by an examination of the mass per unit area vs. thickness plot. This curve should, according to conventional wrought theory, extrapolate somewhat linearly through zero. This curve in Figure 10 clearly does not. If the data supporting these points is accurate, a sharp discontinuity must exist during the first few micro-inches of deposition. This would imply a very low density factor for very thin films which would increase until about 20 or 30 micro-inches, whereupon it would stabilize to a linear plot possibly parallel to, but displaced from wrought characteristics.

From the limited extent of this study, it appears that, assuming similar evaporation processes, the materials of either lighter molecular weight, or higher chemical activity suffer most severely from process variations in maintaining the resistivity and surface mass relationships with thickness that conventional bulk theory would imply.

The implications of the relatively few samples studied here would require considerable control to be exercised in the deposition process before any aluminum thickness determinations could be considered by resistance or mass dependent techniques.

The gold and copper, possessing different physical or chemical characteristics, could be satisfactorily measured by either the mass or the resistive principles.

The microtome thickness measurements of the aluminum are of interest in that the vendor claimed to be sending three rolls of aluminized mylar or different thickness. Either our microscope measurements or his methods are in question. The microscope techniques used in this program are more or less substantiated by their repeatability on any given specimen's measurement and their relatively good correlation with the other physical characteristics.

In general, it is the opinion of the writer, based on this work, that thickness measurements of aluminum should be made directly by methods similar to the microtome technique described in this work, or by other direct mechanical or optical means. The mechanical methods would be encumbered by the direct physical contact required and the finite pressure which must exist.

A brief attempt was made to obtain a thickness measurement directly using interferometry. Several samples of gold, aluminum and copper were electro-etched to give a sloped step function across the specimen. Although the slope was gradual enough, the mylar exhibited a severe grainyness when pressured against an optical flat. This prevented any readable fringe pattern from forming for a reading. If this type of accurate calibration is ever needed, it is recommended that heavy flat plastic specimens be metalized concurrently with the mylar. Heavy plastic is recommended over glass flats due to its similarity of Beta scattering cross section to the mylar; the glass having a significantly higher equivalent atomic number.

5.0 ACKNOWLEDGEMENTS

Sincerest appreciation is extended to the following companies and their local representatives for their cooperation and help in allowing the use of their products and equipment for this work:

Hastings and Company for their samples of metalized mylar.

The Schjeldahl Company for their samples of metalized mylar.

Unit Process Corp. and Quality Control Co. for the use of their Microderm Beta Gage.

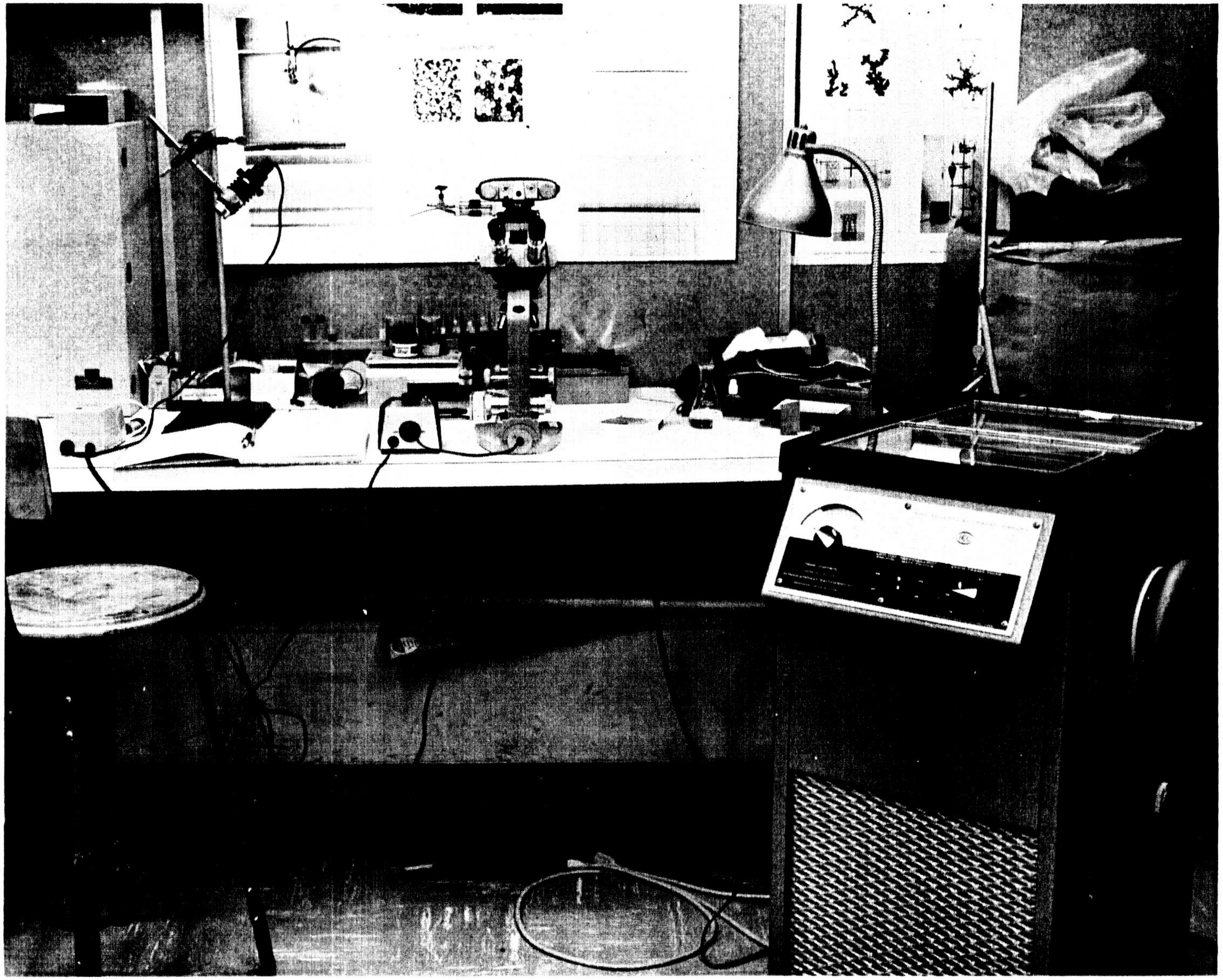
International Equipment Co. and Scientific Products for the use of the Microtome.

The Instrument Division of the Budd Co. for their Radac 210 measurement.

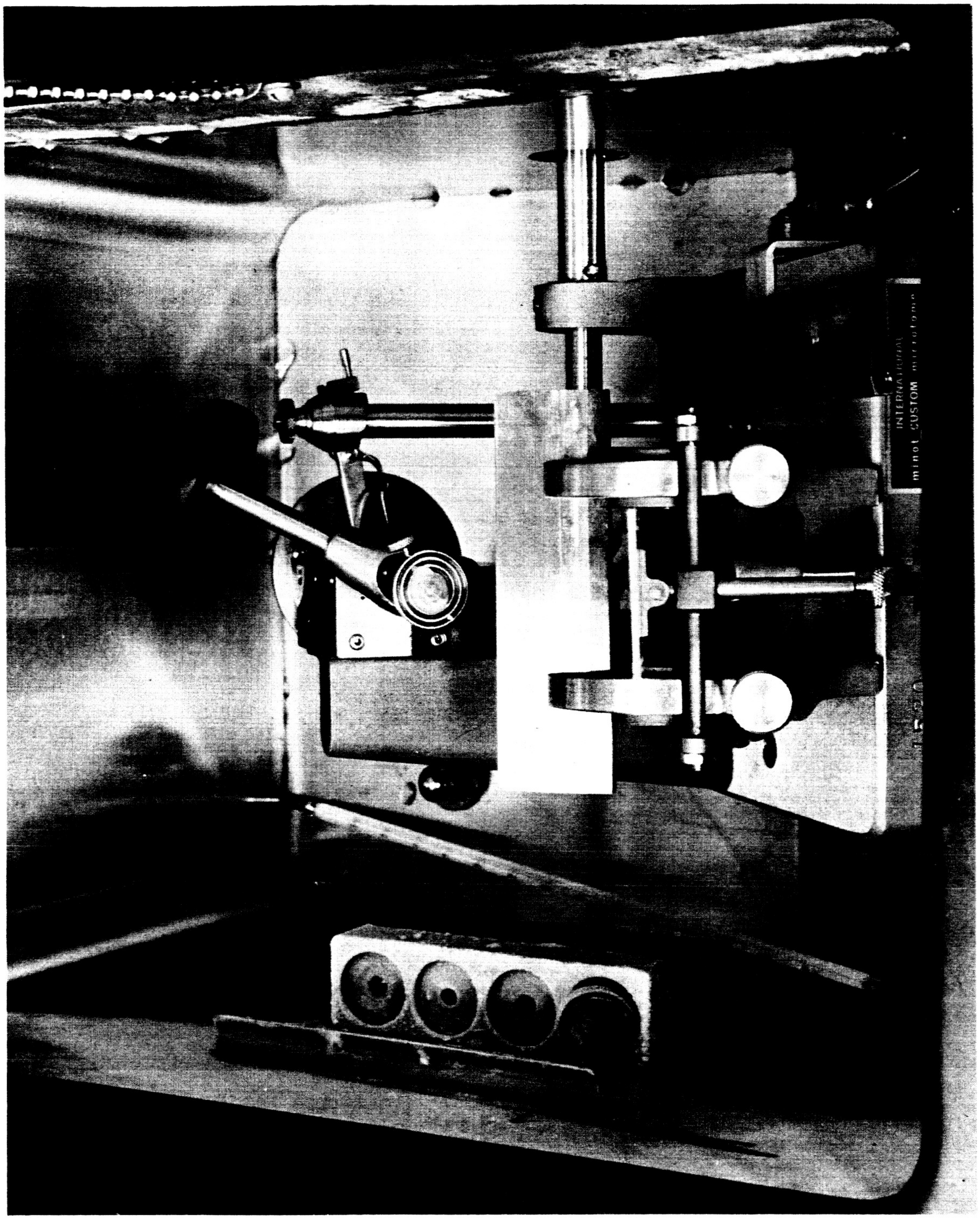
6.0 DISTRIBUTION

National Aeronautics and Space Administration - 1 reproducible/10 copies

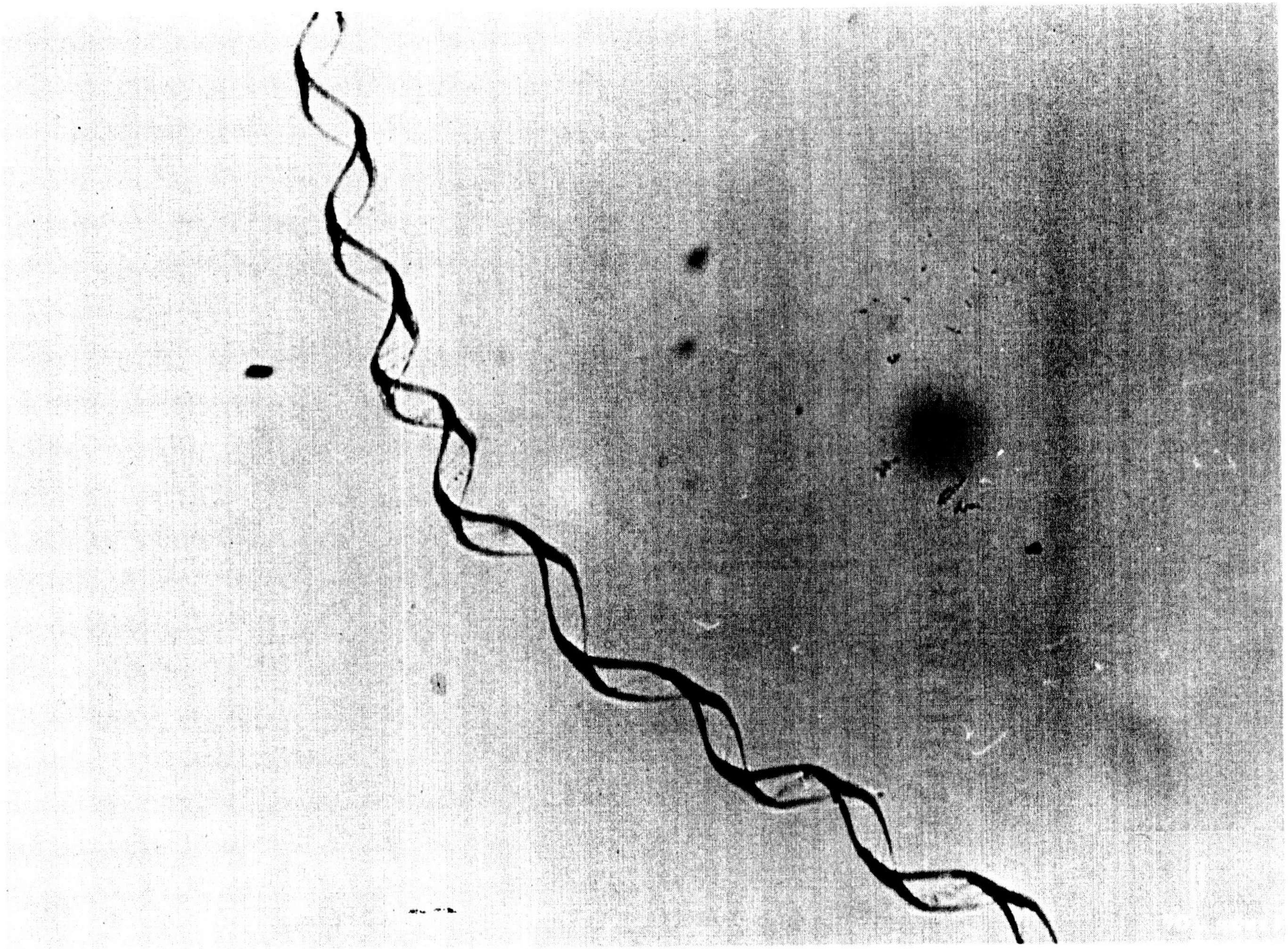
The Hastings Co. -	2 copies
The Schjeldahl Co. -	2 copies
Unit Process Corp. -	1 copy
The Budd Co. -	2 copies
Space-General Corp. -	5 copies and original
Scientific Products -	1 copy



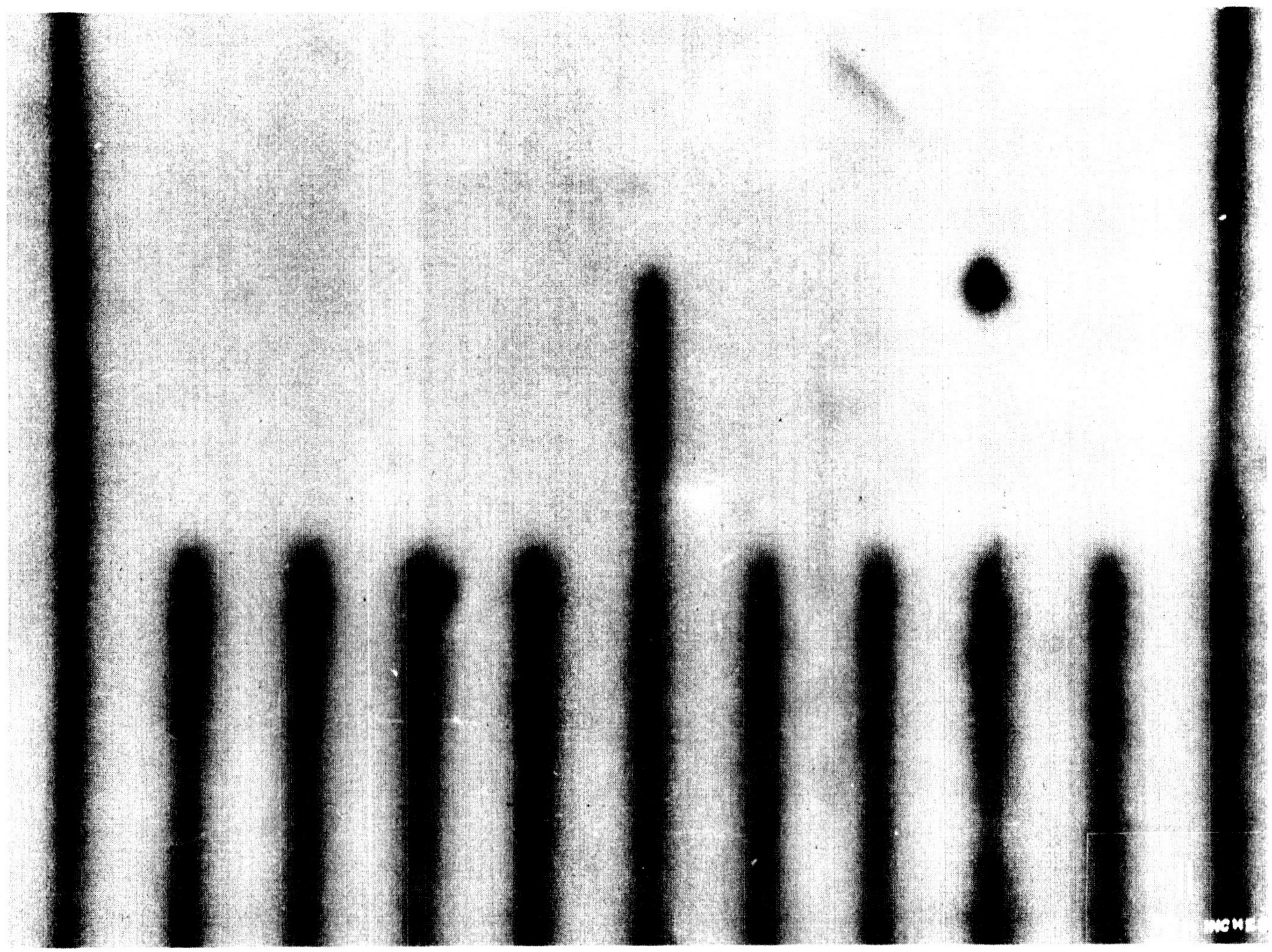
Work Station Showing Microscope and Microtome Used



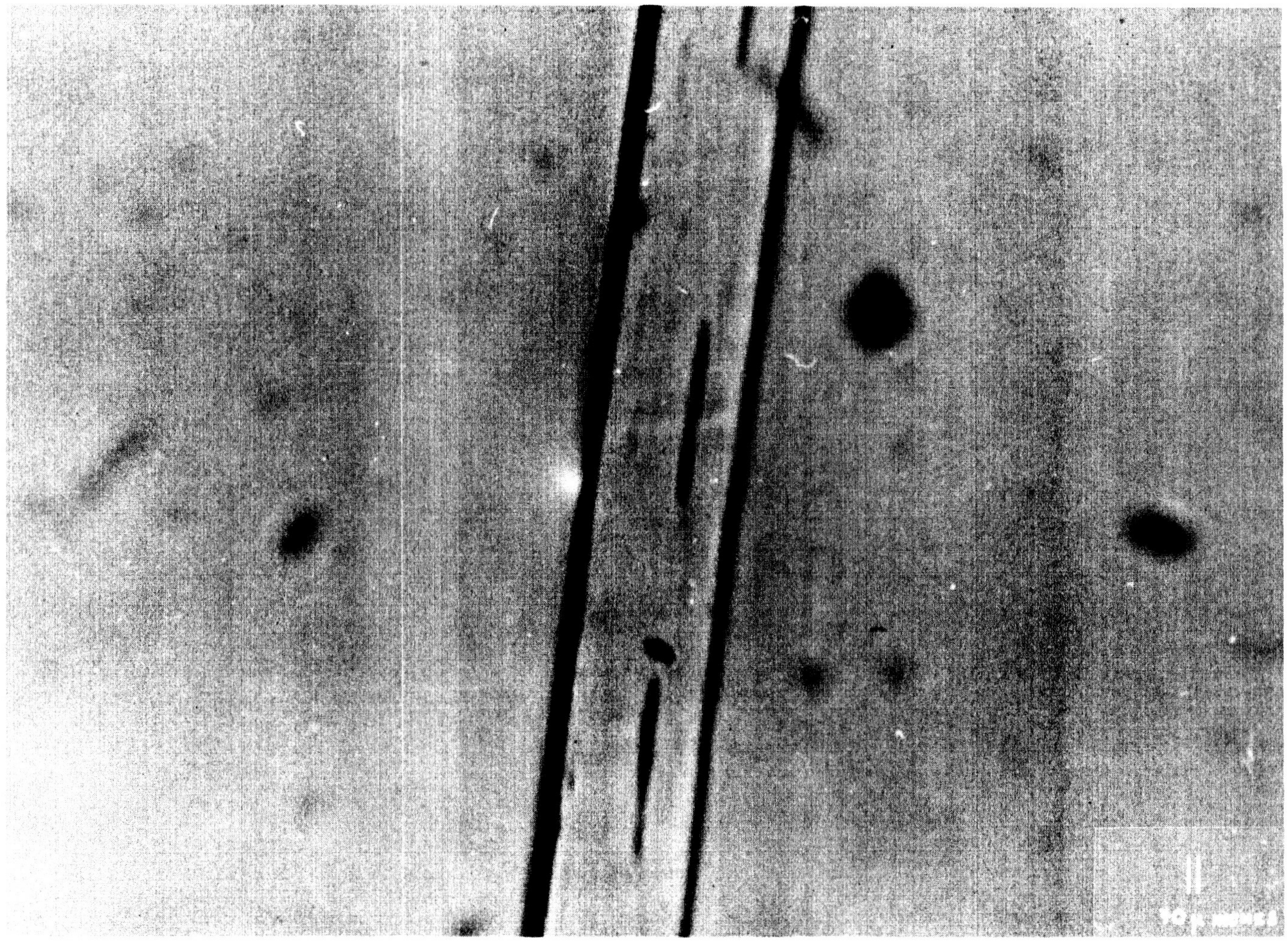
Inside View of Microtome Machine Showing Sample Ready for Slicing



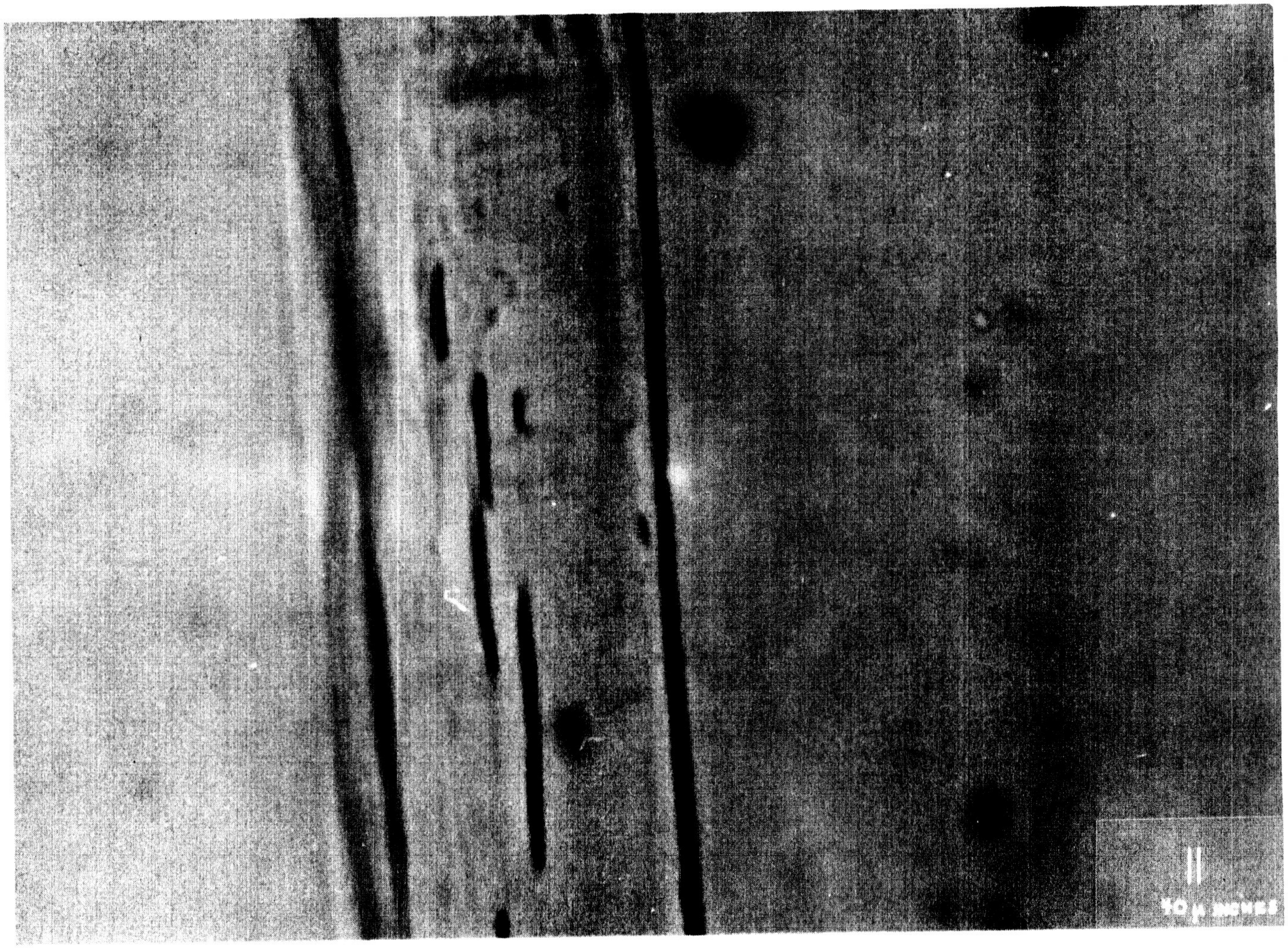
Example of Spiraling When Sample is not Parallel to Microtome Blade



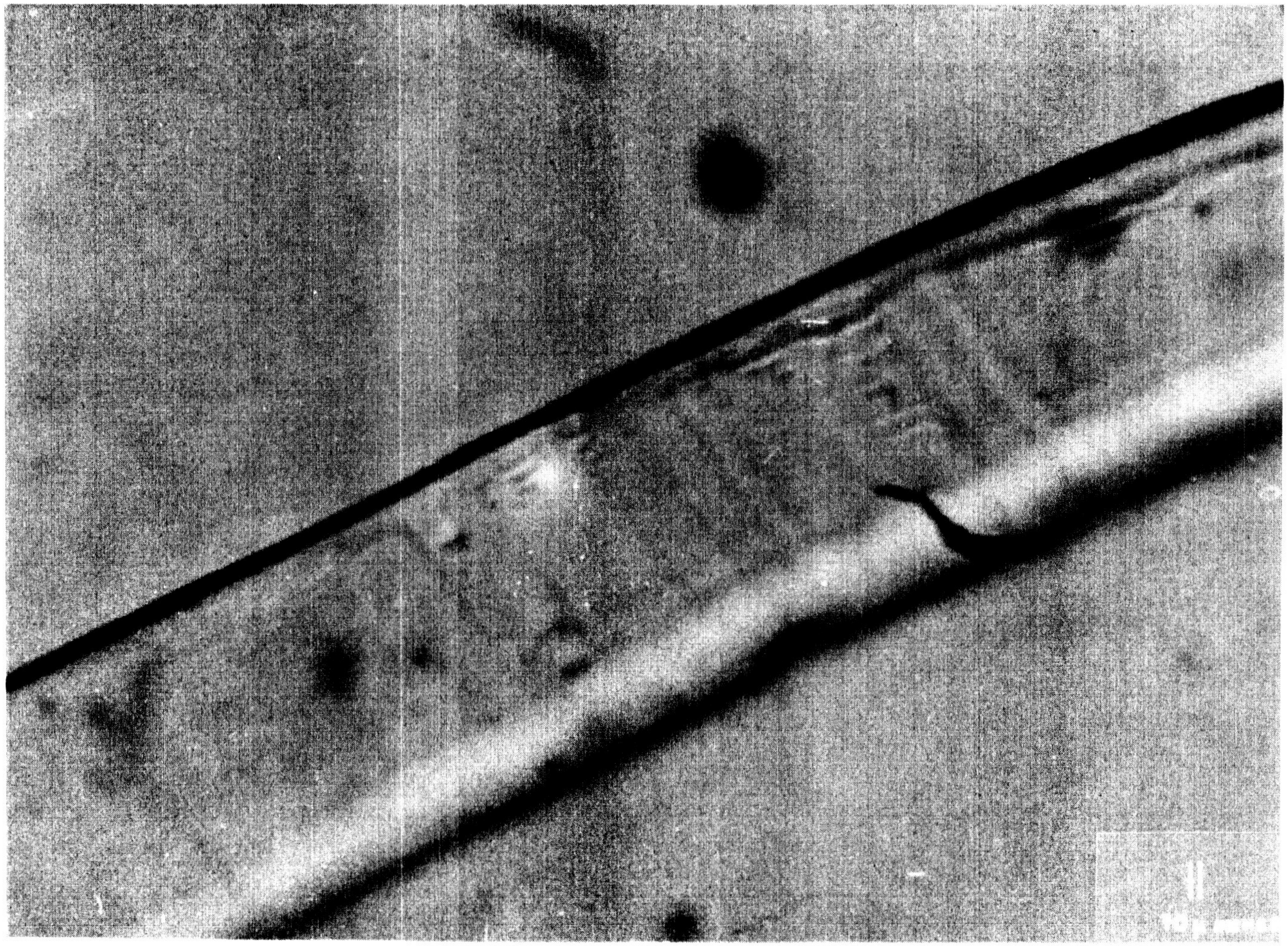
Stage Micrometer   1 division = .01 mm



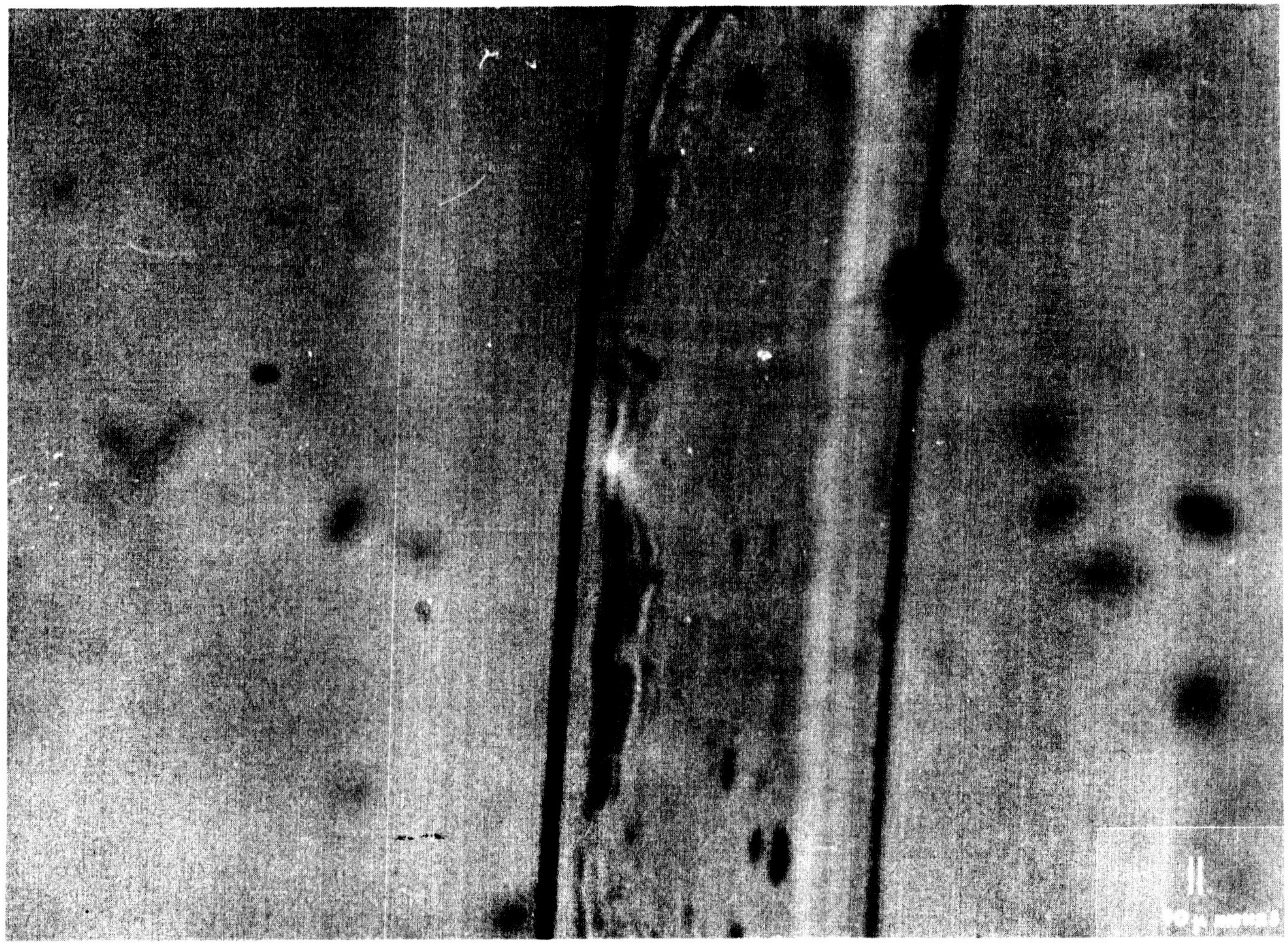
Typical 2AZ cross section



Typical 3AZ cross section

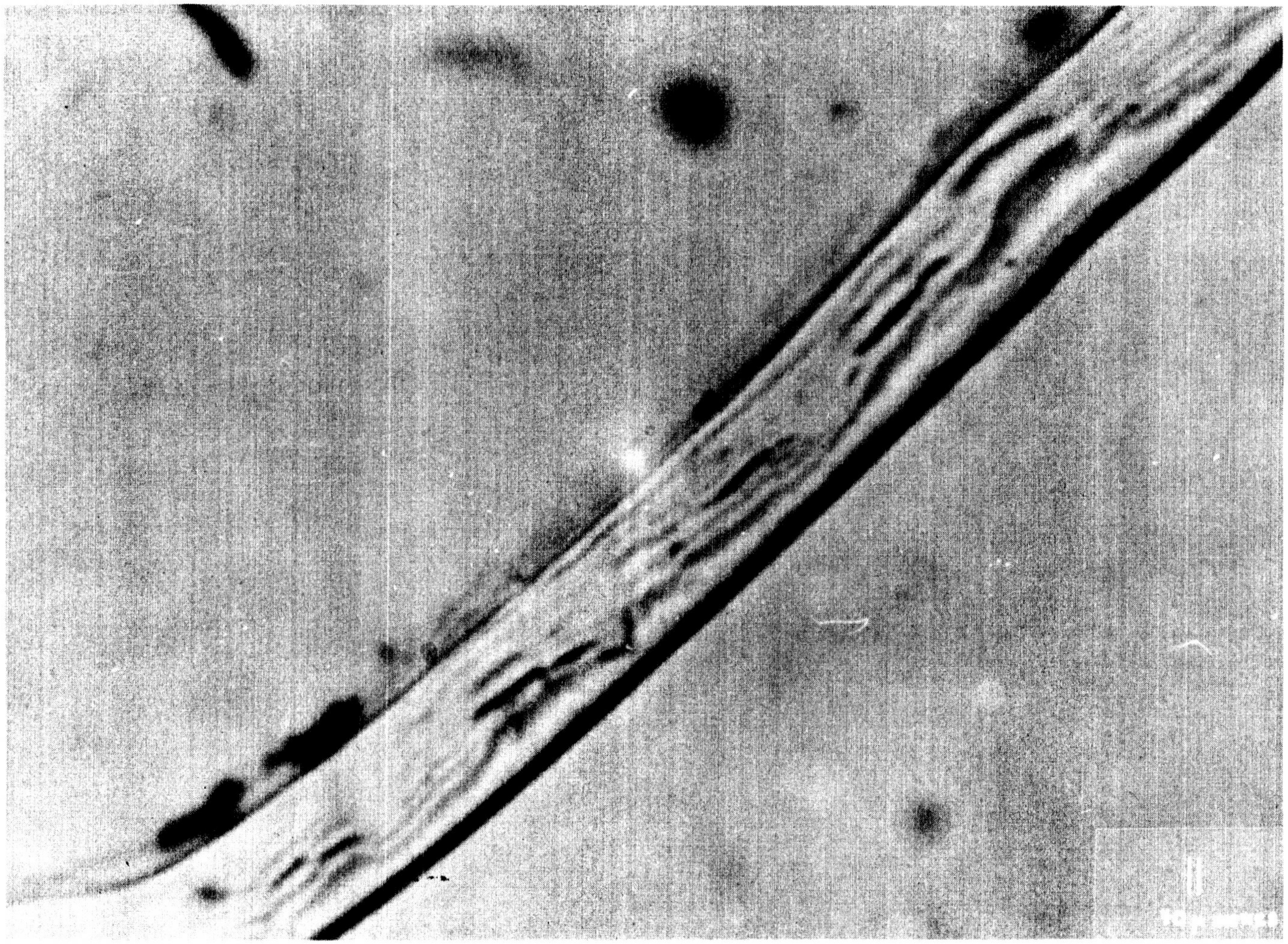


Typical 4AZ cross section

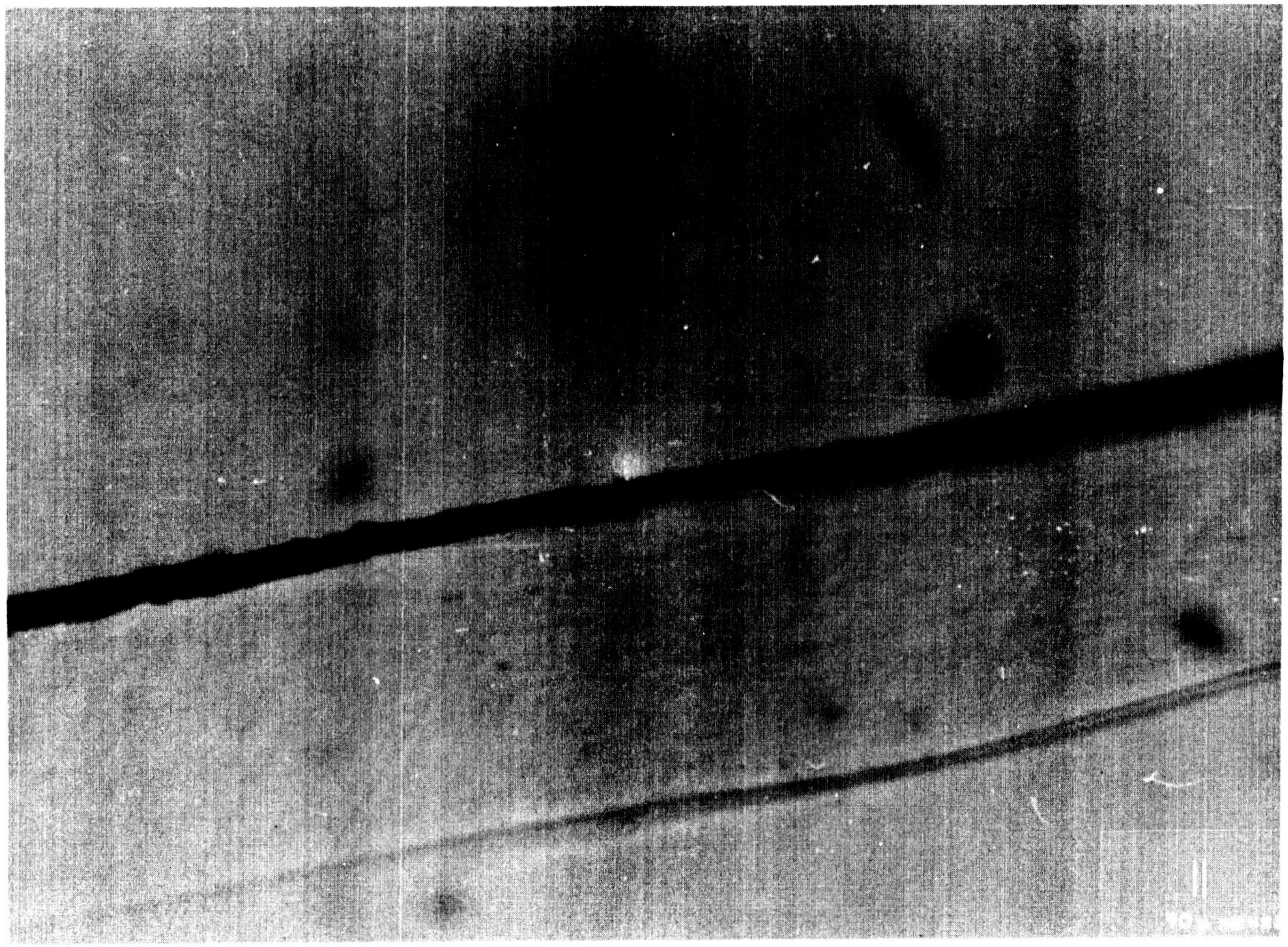


Typical 5AZ cross section

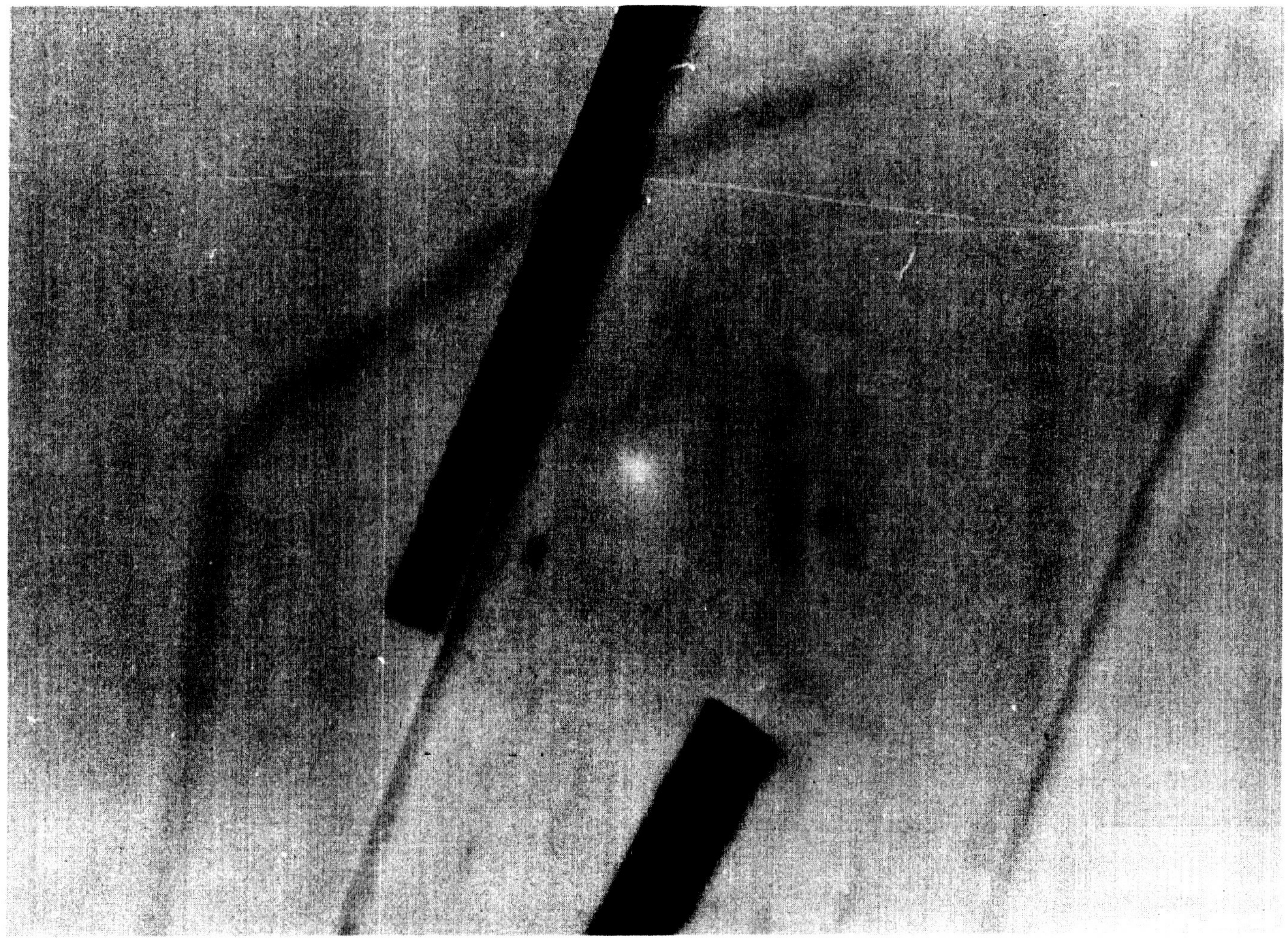
Typical 6GZ cross section



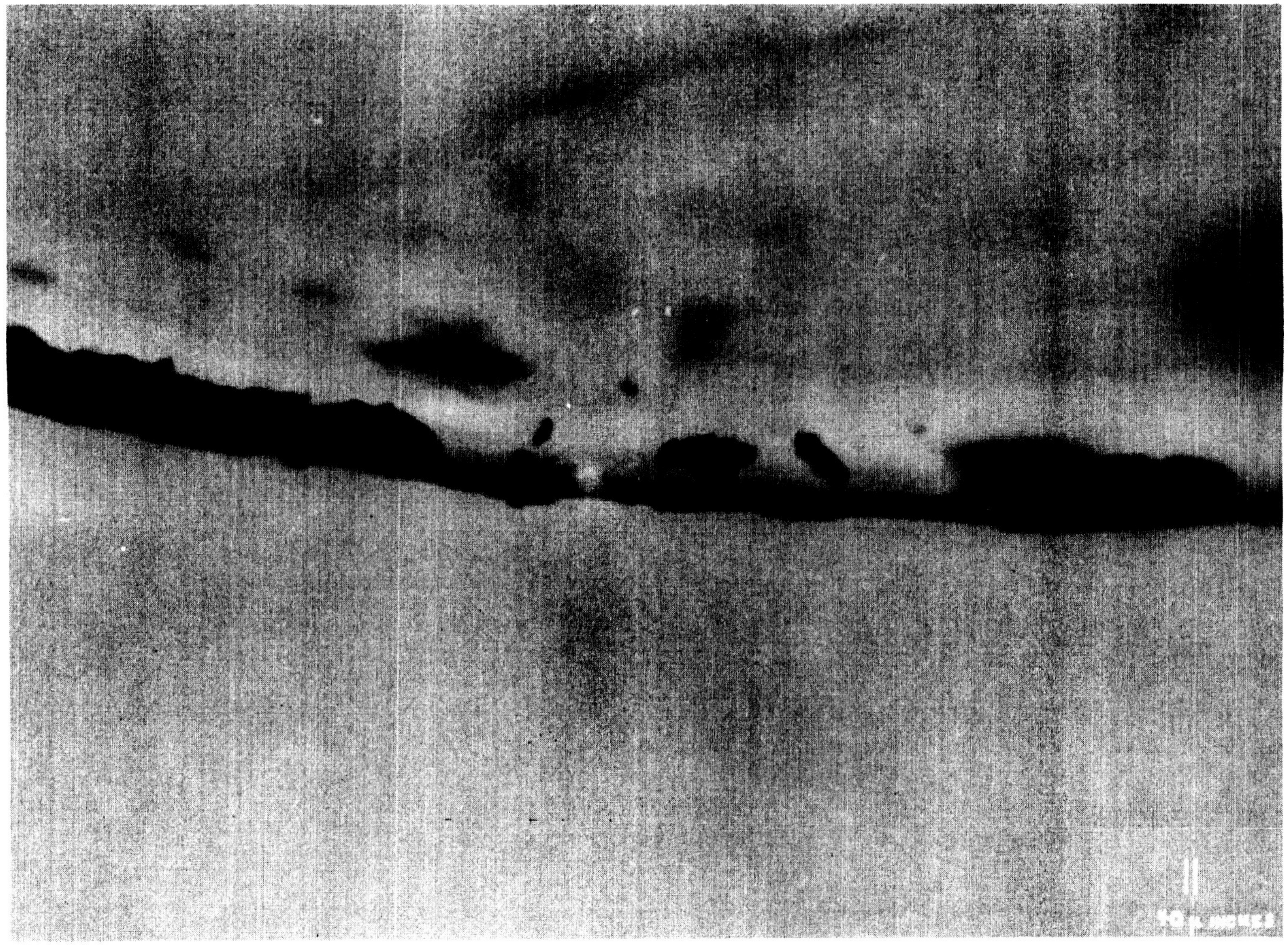
Typical 7GZ cross section



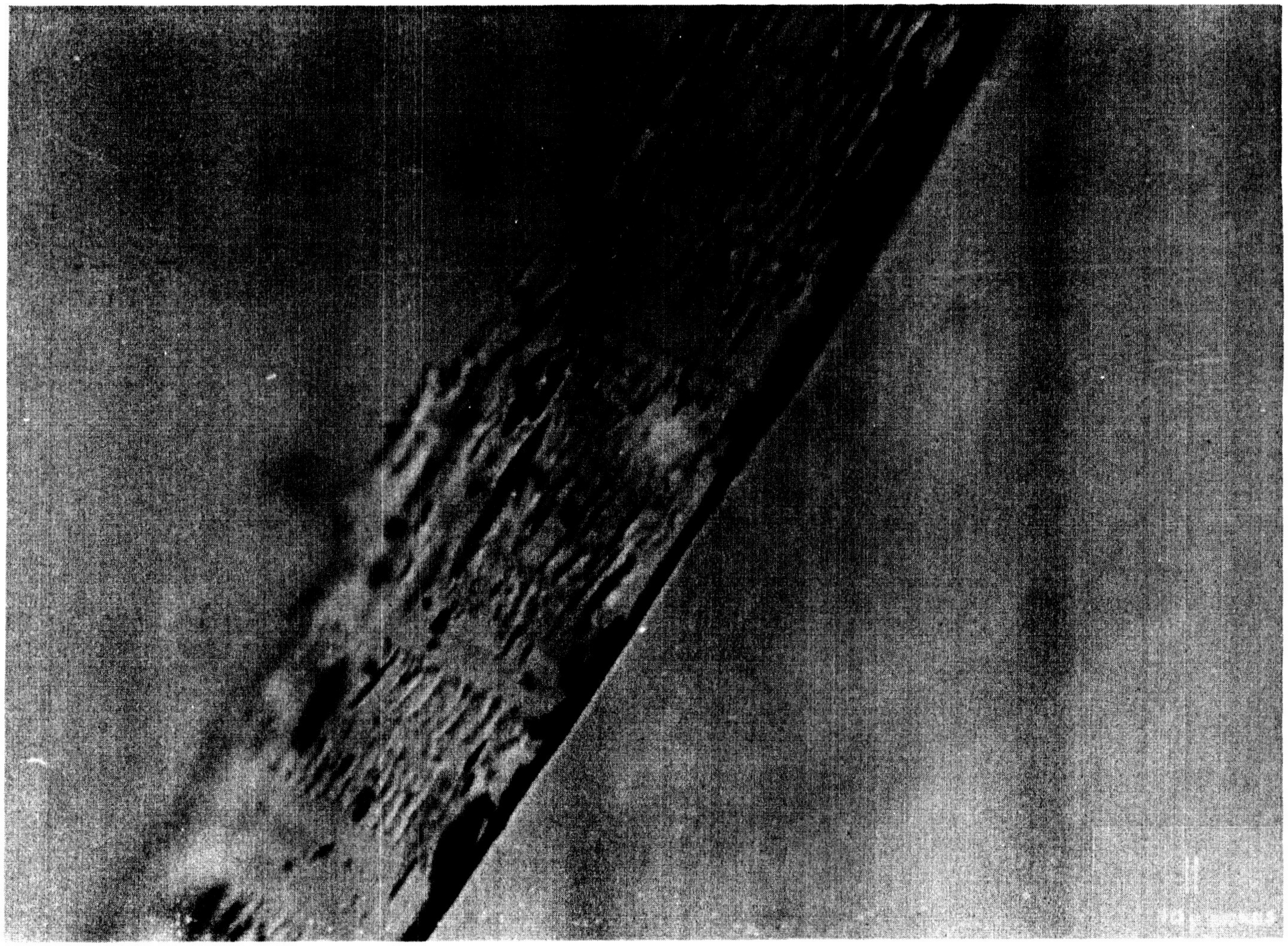
Typical 8GZ cross section



Typical 10CZ cross section



Typical 11CZ cross section



Typical 12CZ cross section